

10/751,314

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NEWS 8 DEC 15 MEDLINE update schedule for December 2004  
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alerts (SDIs) affected  
NEWS 10 DEC 17 COMPUAB reloaded; updating to resume; current-awareness  
alerts (SDIs) affected  
NEWS 11 DEC 17 SOLIDSTATE reloaded; updating to resume; current-awareness  
alerts (SDIs) affected  
NEWS 12 DEC 17 CERAB reloaded; updating to resume; current-awareness  
alerts (SDIs) affected  
NEWS 13 DEC 17 THREE NEW FIELDS ADDED TO IFIPAT/IFIUDB/IFICDB  
NEWS 14 DEC 30 EPFULL: New patent full text database to be available on STN  
NEWS 15 DEC 30 CAPLUS - PATENT COVERAGE EXPANDED  
NEWS 16 JAN 03 No connect-hour charges in EPFULL during January and  
February 2005  
NEWS 17 JAN 11 CA/CAPLUS - Expanded patent coverage to include Russia  
(Federal Institute of Industrial Property)  
  
NEWS EXPRESS JANUARY 10 CURRENT WINDOWS VERSION IS V7.01a, CURRENT  
MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP),  
AND CURRENT DISCOVER FILE IS DATED 10 JANUARY 2005  
  
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FILE 'HOME' ENTERED AT 14:47:55 ON 23 JAN 2005

=> fil reg

COST IN U.S. DOLLARS

SINCE FILE

TOTAL

ENTRY

SESSION

FULL ESTIMATED COST

0.21

0.21

FILE 'REGISTRY' ENTERED AT 14:48:09 ON 23 JAN 2005

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STRUCTURE FILE UPDATES: 21 JAN 2005 HIGHEST RN 818374-94-6

DICTIONARY FILE UPDATES: 21 JAN 2005 HIGHEST RN 818374-94-6

TSCA INFORMATION NOW CURRENT THROUGH MAY 21, 2004

Please note that search-term pricing does apply when conducting SmartSELECT searches.

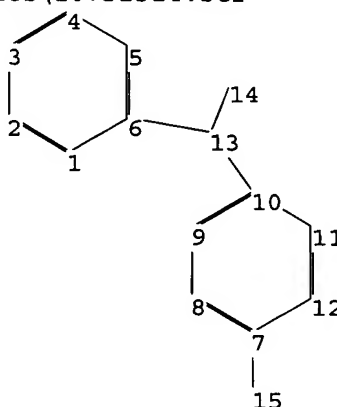
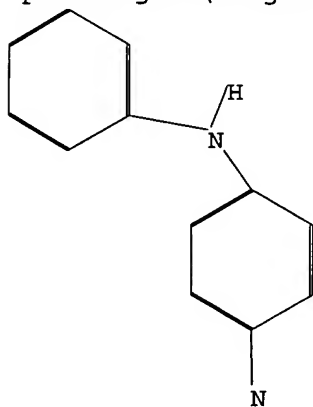
Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. For more information enter HELP PROP at an arrow prompt in the file or refer to the file summary sheet on the web at:

<http://www.cas.org/ONLINE/DBSS/registryss.html>

=>

Uploading C:\Program Files\Stnexp\Queries\10751314.str



chain nodes :

13 14 15

ring nodes :

1 2 3 4 5 6 7 8 9 10 11 12

chain bonds :

6-13 7-15 10-13 13-14

ring bonds :

1-2 1-6 2-3 3-4 4-5 5-6 7-8 7-12 8-9 9-10 10-11 11-12

exact/norm bonds :

6-13 7-15 10-13

exact bonds :

13-14

normalized bonds :

1-2 1-6 2-3 3-4 4-5 5-6 7-8 7-12 8-9 9-10 10-11 11-12

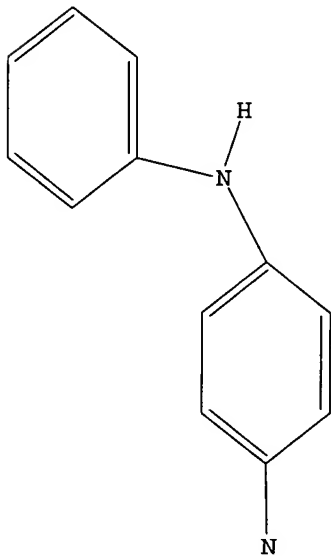
Match level :

1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:Atom 8:Atom 9:Atom 10:Atom  
11:Atom 12:Atom 13:CLASS 14:CLASS 15:CLASS

L1 STRUCTURE UPLOADED

=> d query

L1 STR



Structure attributes must be viewed using STN Express query preparation.

=> s l1

SAMPLE SEARCH INITIATED 14:48:35 FILE 'REGISTRY'  
SAMPLE SCREEN SEARCH COMPLETED - 4544 TO ITERATE

22.0% PROCESSED 1000 ITERATIONS  
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)  
SEARCH TIME: 00.00.01

50 ANSWERS

FULL FILE PROJECTIONS: ONLINE \*\*COMPLETE\*\*  
BATCH \*\*COMPLETE\*\*  
PROJECTED ITERATIONS: 86838 TO 94922  
PROJECTED ANSWERS: 22958 TO 27206

L2 50 SEA SSS SAM L1

=> s l1 full

FULL SEARCH INITIATED 14:48:39 FILE 'REGISTRY'  
FULL SCREEN SEARCH COMPLETED - 91482 TO ITERATE

100.0% PROCESSED 91482 ITERATIONS  
SEARCH TIME: 00.00.01

26808 ANSWERS

L3 26808 SEA SSS FUL L1

=> fil caplus  
COST IN U.S. DOLLARS  
FULL ESTIMATED COST

SINCE FILE	TOTAL
ENTRY	SESSION
161.33	161.54

FILE 'CAPLUS' ENTERED AT 14:48:44 ON 23 JAN 2005  
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FILE COVERS 1907 - 23 Jan 2005 VOL 142 ISS 5  
FILE LAST UPDATED: 21 Jan 2005 (20050121/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s l3  
L4 21526 L3

=> s cu or copper  
771488 CU  
4612 CUS  
773481 CU  
(CU OR CUS)  
830658 COPPER  
421 COPPERS  
830723 COPPER  
(COPPER OR COPPERS)  
L5 1107313 CU OR COPPER

=> s l4 and l5  
L6 1545 L4 AND L5

=> s catalyst  
678330 CATALYST  
681584 CATALYSTS  
L7 869159 CATALYST  
(CATALYST OR CATALYSTS)

=> s l6 and l7  
L8 234 L6 AND L7

=> s ?aniline  
L9 165868 ?ANILINE

=> s aniline  
95112 ANILINE  
12252 ANILINES  
L10 99949 ANILINE

(ANILINE OR ANILINES)

=> s 19 or 110

L11 169029 L9 OR L10

=> s 18 and 111

L12 86 L8 AND L11

=> d 112 1-86 abs ibib

L12 ANSWER 1 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Immobilization of copper onto modified Wang resin provided a polymer-supported copper catalyst, which is effective in cross-coupling reactions between N- or O-containing substrates and arylboronic acids. The copper catalyst is air stable and can be recycled with minimal loss of activity.

ACCESSION NUMBER: 2004:650379 CAPLUS  
 DOCUMENT NUMBER: 141:331857  
 TITLE: Polymer-Supported Copper Complex for C-N and C-O Cross-Coupling Reactions with Aryl Boronic Acids  
 AUTHOR(S): Chiang, Gary C. H.; Olsson, Thomas  
 CORPORATE SOURCE: AstraZeneca R&D Moelndal, Moelndal, SE-431 83, Swed.  
 SOURCE: Organic Letters (2004), 6(18), 3079-3082  
 CODEN: ORLEFF; ISSN: 1523-7060  
 PUBLISHER: American Chemical Society  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 REFERENCE COUNT: 36 THERE ARE 36 CITED REFERENCES AVAILABLE FOR THIS  
 RECORD. ALL CITATIONS AVAILABLE IN THE RE

FORMAT

L12 ANSWER 2 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Cu(I) complexes with phosphorus-containing ligands are claimed as coupling catalysts. For example, CuLBr (L = (2-di-tert-butylphosphino)biphenyl) was prepared from CuBr and L in MeOH. CuLBr was used as a coupling catalyst in presence of K2CO3 for p-bromoacetophenone and n-octanethiol giving p-(octylthio)acetophenone in 80% yield.

ACCESSION NUMBER: 2004:568196 CAPLUS  
 DOCUMENT NUMBER: 141:115950  
 TITLE: Copper complexes of phosphorus-containing ligands and their use as coupling catalysts  
 INVENTOR(S): Scholz, Ulrich; Kunz, Klaus; Gaertzen, Oliver; Benet-Buchholz, Jordi; Wesener, Joachim  
 PATENT ASSIGNEE(S): Bayer Chemicals AG, Germany  
 SOURCE: Eur. Pat. Appl., 17 pp.  
 CODEN: EPXDXW  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

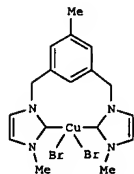
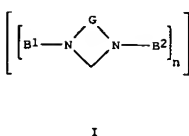
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1437356	A1	20040714	EP 2003-29975	20031230
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
DE 10300097	A1	20040722	DE 2003-10300097	20030107
US 2004198997	A1	20041007	US 2004-752413	20040106
PRIORITY APPLN. INFO.:			DE 2003-10300097	A 20030107

OTHER SOURCE(S): MARPAT 141:115950  
 REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS  
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FORMAT

L12 ANSWER 3 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 GI

L12 ANSWER 3 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

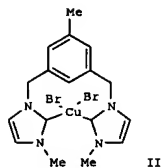
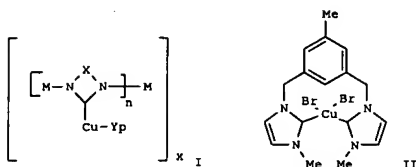


AB The preparation of copper carbene complexes I (G = 1,2-ethanediyl, etc.; B1 = N or S hetero atom containing C5-18 aryl, C1-18 alkyl, C6-19 aralkyl, etc.; B2 = C2-40 organo, etc.; n = 1-3) and their use as coupling reaction catalysts is described. Thus, reaction of 1,3-bis-N-(N-methylimidazolium)methyl-5-methylbenzene dichloride with KOBu-t at 0° in PhMe followed by treatment with CuBr2 gave title compound II which was used as coupling reaction catalyst for 3-(F3C)C6H4I with octanethiol.

ACCESSION NUMBER: 2004:568195 CAPLUS  
 DOCUMENT NUMBER: 141:123760  
 TITLE: Preparation of copper carbene complexes and their use as coupling reaction catalysts  
 INVENTOR(S): Kunz, Klaus; Scholz, Ulrich; Gaertzen, Oliver; Ganzer, Dirk; Wesener, Joachim  
 PATENT ASSIGNEE(S): Bayer Chemicals AG, Germany  
 SOURCE: Eur. Pat. Appl., 20 pp.  
 CODEN: EPXDXW  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1437355	A1	20040714	EP 2003-29974	20031230
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
DE 10300098	A1	20040715	DE 2003-10300098	20030107
US 2004192664	A1	20040930	US 2004-752353	20040106
PRIORITY APPLN. INFO.:			DE 2003-10300098	A 20030107

OTHER SOURCE(S): CASREACT 141:123760; MARPAT 141:123760  
 REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS  
 RECORD. ALL CITATIONS AVAILABLE IN THE RE



AB Copper catalysts I [X = CH<sub>2</sub>CH<sub>2</sub>, CH:CH; M = alkyl, aralkyl, aryl, heteroaryl; the two substituents M may be linked via an alkylidene, arylidyl, or heteroarylidyl bridge; Y = halogen, CF<sub>3</sub>CO, CF<sub>3</sub>SO<sub>2</sub>, F<sub>3</sub>C(CF<sub>2</sub>)<sub>3</sub>SO<sub>2</sub>, CN, Ac, fluoroacetylacetonyl, NO<sub>2</sub>, arylsulfonyl, oxinate, phosphate, carbonate, BF<sub>4</sub>; n = 1-3; x = 1-6; q = 0-2] were prepared

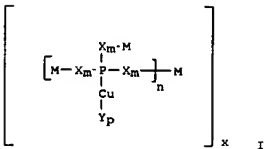
for use in the coupling reaction between anilines and halonitrobenzenes to form nitrodiphenylamines. Thus, the catalyst II was prepared by treating the bisimidazolium salt with CuBr<sub>2</sub> and was

used to couple PhNH<sub>2</sub> with 4-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>Cl to give PhNH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>-4 which was reduced to PhNH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>-4.

ACCESSION NUMBER: 2004:568190 CAPLUS  
DOCUMENT NUMBER: 141:106260  
TITLE: Process for the preparation of nitrodiphenylamines  
INVENTOR(S): Kunz, Klaus; Haider, Joachim; Ganzer, Dirk; Scholz, Ulrich; Sicheneder, Adolf  
PATENT ASSIGNEE(S): Bayer Aktiengesellschaft, Germany  
SOURCE: Eur. Pat. Appl., 12 pp.  
CODEN: EPXXDW  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1437341	A1	20040714	EP 2003-29846	20031224
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
DE 10300125	A1	20040715	DE 2003-10300125	20030107
US 2004143138	A1	20040722	US 2004-751311	20040102
US 6815562	B2	20041109		
JP 2004210791	A2	20040729	JP 2004-1698	20040107
PRIORITY APPL. INFO.:			DE 2003-10300125	A 20030107

OTHER SOURCE(S): MARPAT 141:106260  
REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE  
FORMAT



AB Aminodiphenylamines are prepared by reaction of nitrohalobenzenes with anilines in presence of a Cu catalyst I [X = O, NH, S, CH<sub>2</sub>, CH<sub>2</sub>CH<sub>2</sub>, (CH<sub>2</sub>)<sub>3</sub>; M = aryl, aralkyl, alkyl, heteroaryl and two M residues may be linked by an alkylene bridge; Y = halogen, CF<sub>3</sub>CO, CF<sub>3</sub>SO<sub>2</sub>, F<sub>3</sub>C(CF<sub>2</sub>)<sub>3</sub>SO<sub>2</sub>, CN, Ac, acetylacetonyl, fluoroacetylacetonyl, NO<sub>2</sub>, arylsulfonyl, oxinate, phosphate, carbonate, BF<sub>4</sub>; n = 0, 1; n = 1-3; p = 0-2; x = 1-6], followed by hydrogenation. Thus, PhNH<sub>2</sub> was treated with 4-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>Cl in presence of CuBr, (Me<sub>3</sub>C)P(C<sub>6</sub>H<sub>4</sub>)Ph-2, K<sub>2</sub>CO<sub>3</sub> to give 41.2% 4-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHPh which was reduced to 4-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHPh with Raney Ni.

ACCESSION NUMBER: 2004:568189 CAPLUS  
DOCUMENT NUMBER: 141:106259  
TITLE: Process for the preparation of aminodiphenylamines  
INVENTOR(S): Haider, Joachim; Scholz, Ulrich; Sicheneder, Adolf  
PATENT ASSIGNEE(S): Bayer Aktiengesellschaft, Germany  
SOURCE: Eur. Pat. Appl., 12 pp.  
CODEN: EPXXDW  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1437340	A1	20040714	EP 2003-29845	20031224
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
DE 10300126	A1	20040715	DE 2003-10300126	20030107
US 2004143139	A1	20040722	US 2004-751314	20040102
JP 2004210787	A2	20040729	JP 2004-1261	20040106
PRIORITY APPL. INFO.:			DE 2003-10300126	A 20030107

OTHER SOURCE(S): MARPAT 141:106259  
REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE  
FORMAT

L12 ANSWER 6 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
AB An exptl. simple microwave-assisted solvent-free N-arylation of primary amines with sodium tetraphenylborate or arylboronic acids, promoted by inexpensive cupric acetate, on the surface of KF-alumina, is reported. The reaction was selective for mono-N-arylation, and a variety of functional groups were tolerated in the process.

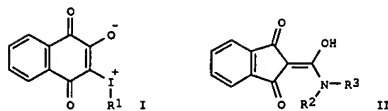
ACCESSION NUMBER: 2004:509896 CAPLUS  
DOCUMENT NUMBER: 141:140119  
TITLE: Microwave-assisted copper-promoted N-arylation of amines with aryl boronic acids/salts on a KF-alumina surface  
AUTHOR(S): Das, Pralay; Basu, Basudeb  
CORPORATE SOURCE: Department of Chemistry, North Bengal University, Darjeeling, India  
SOURCE: Synthetic Communications (2004), 34(12), 2177-2184  
CODEN: SYNGAV; ISSN: 0039-7911  
PUBLISHER: Marcel Dekker, Inc.  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
OTHER SOURCE(S): CASREACT 141:140119  
REFERENCE COUNT: 33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE  
FORMAT

L12 ANSWER 7 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB 4-Alkenyl-4'-nitrodiphenylamines are produced by reacting alkenylanilines with nitrohalobenzenes in the presence of a Cu or Pd catalyst and a base. The resulting 4-alkenyl-4'-nitrodiphenylamines were reduced to 4-alkenyl-4'-aminodiphenylamines. Thus, 4-isopropenylaniline, palladiumacetylacetonate, and 2,2'-bis(diphenylphosphino)-1,1'-binaphthyl in xylene were stirred under an inert atmosphere for 10 min at room temperature followed by stirring with 4-chloronitrobenzene for 10 min at room temperature and reflux with K<sub>3</sub>PO<sub>4</sub> for 4 h to give 99% 4-isopropenyl-4'-nitrodiphenylamine. A mixture of 4-isopropenyl-4'-nitrodiphenylamine, toluene, and Pd/C was treated with H<sub>2</sub> for 8 h at 25° to give 99% 4-isopropenyl-4'-aminodiphenylamine.  
 ACCESSION NUMBER: 2004:157441 CAPLUS  
 DOCUMENT NUMBER: 140:199102  
 TITLE: Procedure for the production of 4-alkenyl-4'-nitrodiphenylamines and 4-alkenyl-4'-aminodiphenylamines  
 INVENTOR(S): Ooms, Pieter; Scholz, Ulrich; Haider, Joachim  
 PATENT ASSIGNEE(S): Bayer A.-G., Germany  
 SOURCE: Ger. Offen., 7 pp.  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 10235834	A1	20040226	DE 2002-10235834	20020806
PRIORITY APPLN. INFO.:			DE 2002-10235834	20020806

L12 ANSWER 9 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB A polyaniline (PANI) o-PANI-co-p-PANI copolymer was prepared which has solubilizing and functional substituents on the conducting polymer backbone that do not adversely affect the conductivity. An urea-protected aniline trimer macromonomer with para-ortho-para connectivity and bromine end groups underwent regiospecific coupling with a di-BOC-protected aniline trimer comonomer with all para connectivity and amine end groups using Pd catalyst, to obtain 5-p,o-PANI. The 5-p,o-PANI has a repeat unit consisting of five para-substituted aniline rings for every one ortho-substituted aniline ring. The regioregularity of 5-p,o-PANI (M<sub>n</sub> = 14 000, GPC in NMP, polystyrene stds.) was established using <sup>1</sup>H NMR, <sup>13</sup>C(H) NMR, and IR spectroscopy. Analogous with p-PANI, the 5p,o-PANI exhibited three distinct oxidation states. The conductivity of blends of 5-p,o-PANI and PVC was on the same order of magnitude as that of PANI films prepared under the same conditions.  
 ACCESSION NUMBER: 2003:365473 CAPLUS  
 DOCUMENT NUMBER: 139:101499  
 TITLE: o,p-Polyaniline: A New Form of a Classic Conducting Polymer  
 AUTHOR(S): Ward, Rachel E.; Meyer, Tara Y.  
 CORPORATE SOURCE: Department of Chemistry, University of Pittsburgh, Pittsburgh, PA, 15260, USA  
 SOURCE: Macromolecules (2003), 36(12), 4368-4373  
 CODEN: MAMOBX; ISSN: 0024-9297  
 PUBLISHER: American Chemical Society  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 REFERENCE COUNT: 38 THERE ARE 38 CITED REFERENCES AVAILABLE FOR THIS  
 RECORD. ALL CITATIONS AVAILABLE IN THE RE  
 FORMAT

L12 ANSWER 8 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 GI



AB Phenyliodonium ylide of 2-hydroxy-1,4-naphthoquinone I (R<sub>1</sub> = Ph) reacts with amines R<sub>2</sub>NHR<sub>3</sub> (R<sub>2</sub> = H, R<sub>3</sub> = Ph, 4-MeC<sub>6</sub>H<sub>4</sub>, 4-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>, PhCH<sub>2</sub>, PhCH<sub>2</sub>CH<sub>2</sub>, etc.; R<sub>2</sub> = Me, R<sub>3</sub> = Ph) in refluxing dichloromethane to afford good yields of indanedione 2-carboxamides II through a ring-contraction and o,o'-dioxoketene formation reaction. II exist in solution in an unusual enol-amide form. In contrast, copper-catalyzed reaction of ylides I (R<sub>1</sub> = Ph, 4-MeC<sub>6</sub>H<sub>4</sub>) with R<sub>2</sub>NHR<sub>3</sub> affords arylated amines R<sub>1</sub>NHR<sub>2</sub>R<sub>3</sub> and 3-iodo-4-hydroxy-1,2-naphthoquinone.  
 ACCESSION NUMBER: 2003:435929 CAPLUS  
 DOCUMENT NUMBER: 139:149404  
 TITLE: Studies on the Reactivity of Aryliodonium Ylides of 2-Hydroxy-1,4-naphthoquinone: Reactions with Amines  
 AUTHOR(S): Malamidou-Kenikaki, Elizabeth; Spyroudis, Spyros; Tsanakopoulou, Maria  
 CORPORATE SOURCE: Laboratory of Organic Chemistry, Department of Chemistry, University of Thessaloniki, Thessaloniki, 54124, Greece  
 SOURCE: Journal of Organic Chemistry (2003), 68(14), 5627-5631  
 CODEN: JOCEAH; ISSN: 0022-3263  
 PUBLISHER: American Chemical Society  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 139:149404  
 REFERENCE COUNT: 24 THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS  
 RECORD. ALL CITATIONS AVAILABLE IN THE RE  
 FORMAT

L12 ANSWER 10 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB The first general method for the Pd-catalyzed amination of aryl tosylates and benzenesulfonates was developed utilizing ligand 2,4,6-(Me<sub>2</sub>CH)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>C<sub>6</sub>H<sub>4</sub>PCy<sub>2</sub>-2, which belongs to a new generation of biaryl monophosphine ligands. In addition, the new catalyst system enables amidation of aryl arenesulfonates and aqueous amination protocols that do not necessitate the use of cosolvents. The substrate scope has been significantly expanded to include aryl halides containing primary amides and free carboxylic acid groups. In the case of multifunctional substrates, the Pd-catalyzed amination can provide selectivity that is complementary to the Cu-catalyzed C-N bond-forming processes.  
 ACCESSION NUMBER: 2003:344190 CAPLUS  
 DOCUMENT NUMBER: 139:117168  
 TITLE: Expanding Pd-Catalyzed C-N Bond-Forming Processes: The First Amidation of Aryl Sulfonates, Aqueous Amination, and Complementarity with Cu-Catalyzed Reactions  
 AUTHOR(S): Huang, Xiaohua; Anderson, Kevin W.; Zim, Danilo; Jiang, Lei; Klapars, Artis; Buchwald, Stephen L.  
 CORPORATE SOURCE: Department of Chemistry, Massachusetts Institute of Technology, Cambridge, MA, 02139, USA  
 SOURCE: Journal of the American Chemical Society (2003), 125(22), 6653-6655  
 CODEN: JACSAT; ISSN: 0002-7863  
 PUBLISHER: American Chemical Society  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 139:117168  
 REFERENCE COUNT: 22 THERE ARE 22 CITED REFERENCES AVAILABLE FOR THIS  
 RECORD. ALL CITATIONS AVAILABLE IN THE RE  
 FORMAT



L12 ANSWER 11 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Diphenylamines, useful as additives for rubbers and plastics (no data), are prepared by reaction of phenols with (supercrit.) anilines using Group IA, IIA, VIB, VIIB, VIII, or IB metal chlorides as catalysts. Hydroquinone was treated with aniline in the presence of CuCl<sub>2</sub> at 300° for 30 min to give 51% N-phenyl-p-aminophenol and 40% N,N'-diphenyl-p-phenylenediamine.

ACCESSION NUMBER: 2003:257862 CAPLUS  
 DOCUMENT NUMBER: 138:271374  
 TITLE: Preparation of diphenylamines from phenols and anilines  
 INVENTOR(S): Suzuki, Tomoyuki; Goto, Fumisato  
 PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.  
 CODEN: JKKXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

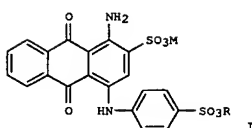
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2003096032	A2	20030403	JP 2001-296463	20010927
PRIORITY APPL. INFO.: JP 2001-296463 20010927				

OTHER SOURCE(S): MARPAT 138:271374

L12 ANSWER 12 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB 6-Amino-5,12-naphthacenedione and 6-amino-5,11-naphthacenedione react with aniline in the presence of cobalt, copper, and manganese salts, resulting in replacement of hydrogen in the peri-position with respect to the carbonyl group by phenylamino group and formation of 11- and 12-phenylamino-6-amino(phenylamino)-5,12- and -5,11-naphthacenequinones.

ACCESSION NUMBER: 2002:282996 CAPLUS  
 DOCUMENT NUMBER: 137:140325  
 TITLE: Direct phenylation of 6-aminonaphthacenequinones promoted by metal salts  
 AUTHOR(S): Sokolyuk, N. T.; Pisulina, L. P.; V'yugin, A. I.  
 CORPORATE SOURCE: Research Institute of Fine Organic Synthesis, "TOS" Joint-Stock Company, Dolgoprudnyi, 141700, Russia  
 SOURCE: Russian Journal of Organic Chemistry (Translation of Zhurnal Organicheskoi Khimii) (2002), 38(1), 70-75  
 CODEN: RJOCEQ; ISSN: 1070-4280  
 PUBLISHER: MAIK Nauka/Interperiodica Publishing  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 137:140325  
 REFERENCE COUNT: 14 THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS  
 RECORD. ALL CITATIONS AVAILABLE IN THE RE  
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L12 ANSWER 13 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
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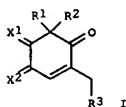
AB Title dyes represented by the general formula I (R = CH<sub>2</sub>CH<sub>2</sub>OSO<sub>3</sub>M or CH:CH<sub>2</sub>, M = H or alkali metal) are obtained by reaction of bromaminic acid or its alkali metal salts and 4-(β-sulfatoethylsulfonyl) aniline or its alkali metal salts in aqueous medium in the presence of phosphate buffers and copper catalysts. Thus, water 48, 54% phosphoric acid 36.3, 28% sodium hydroxide 45.7, 4-(β-sulfatoethylsulfonyl) aniline 30.9, bromaminic acid sodium salt 41.8, and cuprous oxide 5.6 parts were heated at 80° while pH was controlled at 5.8 with sodium carbonate to give a blue dye solution with a ratio of sulfate ester type and vinylsulfone type 38:62 and yield (based on bromaminic acid) 50%, and dye strength 19.7%.

ACCESSION NUMBER: 2002:136608 CAPLUS  
 DOCUMENT NUMBER: 136:87227  
 TITLE: Production method of anthraquinone reactive dyes  
 INVENTOR(S): Tsukise, Bunji; Watanabe, Hitoshi  
 PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.  
 CODEN: JKKXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2002012786	A2	20020115	JP 2000-192351	20000627
PRIORITY APPL. INFO.: JP 2000-192351 20000627				

OTHER SOURCE(S): CASREACT 136:87227; MARPAT 136:87227

L12 ANSWER 14 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
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AB The invention relates to an agent for dyeing keratin containing fibers, especially human hair, comprising at least one cyclopentaquinolalium derivative of the formula (I), wherein R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub> independently represent a hydrogen atom or a C1-C4 alkyl group, and R<sub>1</sub> and R<sub>2</sub> together may form a ring, X<sub>1</sub> and X<sub>2</sub> represent an oxygen or sulfur atom or together a 1,2-arylenediimino group that in turn can be substituted in the aromatic group by halogen atoms, C1-C4 alkyl, C1-C4 alkoxy, nitro, amino, C1-C4 alkyl, C1-C4 alkylamino, hydroxy, carboxy, or sulfo groups or an addnl. condensed aromatic ring and is quaternized on one of the nitrogen atoms by a C1-C6 alkyl, aralkyl, aryl, C2-C4 alkenyl, or C1-C6 hydroxyalkyl, or carboxyalkyl group, and wherein the addnl. condensed aromatic ring may be substituted by halogen atoms, in halogen atoms, C1-C4 alkyl, C1-C4 alkoxy, nitro, amino, C1-C4 alkyl, C1-C4 alkylamino, hydroxy, carboxy, or sulfo groups. The invention further relates to the tautomeric forms or the physiol. acceptable salts of the derivs. Thus 3,3,5-trimethyl-cyclohex-5-en-1,2,4-trione was synthesized from 6-oxo isophorone, using 1,4-dioxane and selenium dioxide catalysts. A dyeing gel contained (g): 3,3,5-trimethyl-cyclohex-5-en-1,2,4-trione 3.3; Natrosol 250 HR 2; water to 100;. The gel was mixed 1:1 with a dyeing cream that contained (g): Texapon NSO 20.00; Dehyton K 12.50; Hydrenol D 8.50; Lorol 2.00; 4,4'-diamino-diphenylene diamine H2SO4 5.95; sodium sulfite 0.10; ascorbic acid 0.10; ammonia (25%) 4.50; water to 100; pH 8.95.

ACCESSION NUMBER: 2001:935366 CAPLUS  
 DOCUMENT NUMBER: 136:74276  
 TITLE: Hair dyeing compositions containing cyclopentaquinolalium derivatives  
 INVENTOR(S): Oberkobusch, Doris; Hoeffkes, Horst; Moeller, Hinrich  
 PATENT ASSIGNEE(S): Gross, Wibke; Martin, Hans-Dieter  
 SOURCE: Henkel Kommanditgesellschaft auf Aktien, Germany  
 PCT Int. Appl., 34 pp.  
 CODEN: PIXXD2  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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L12 ANSWER 14 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)  
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WO 2001097764 A2 20011227 WO 2001-EP6690 20010613  
WO 2001097764 A3 20020523  
W: AU, JP, US  
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL,  
PT, SE, TR  
DE 10029384 A1 20020103 DE 2000-10029384 20000621  
EP 1292268 A2 20030319 EP 2001-943502 20010613  
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,  
IE, FI, CY, TR  
PRIORITY APPLN. INFO.: DE 2000-10029384 A 20000621  
WO 2001-EP6690 W 20010613  
OTHER SOURCE(S): MARPAT 136:74276

L12 ANSWER 15 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
AB 4-Nitrodiphenylamine was synthesized from p-chloronitrobenzene and  
aniline using phase-transfer catalysis. The optimum operation  
conditions and purification methods were found. The true yield and  
purity were  
over 94% and 99%, resp.  
ACCESSION NUMBER: 2001:470307 CAPLUS  
DOCUMENT NUMBER: 136:136580  
TITLE: Synthesis technology of 4-nitrodiphenylamine  
AUTHOR(S): Wei, Qing; Li, Gui-ling; Dai, Zi-ling  
CORPORATE SOURCE: Guangzhou Research Institute of Non-ferrous Metals,  
Canton, 51065, Peop. Rep. China  
SOURCE: Jingxi Huagong Zhongjianti (2001), 31(1), 26-28  
CODEN: JHZZIAR; ISSN: 1009-9212  
PUBLISHER: Jingxi Huagong Zhongjianti Zazhishe  
DOCUMENT TYPE: Journal  
LANGUAGE: Chinese

L12 ANSWER 16 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
AB A general catalytic coupling of arylboronic acids and amines is reported.  
This room-temperature coupling was realized through the use of catalytic  
copper(II) acetate, 2,6-lutidine as base, and myristic acid as an  
additive. Functionalized aniline substrates provided the  
diarylamine coupling products in good yield (58-91%). A variety of  
alkylamines were also successfully coupled to give N-alkyl  
anilines in moderate yield (50-64%).  
ACCESSION NUMBER: 2001:387340 CAPLUS  
DOCUMENT NUMBER: 135:137247  
TITLE: Copper-Catalyzed Coupling of Arylboronic  
Acids and Amines  
AUTHOR(S): Antilla, Jon C.; Buchwald, Stephen L.  
CORPORATE SOURCE: Department of Chemistry, Massachusetts Institute of  
Technology, Cambridge, MA, 02139, USA  
SOURCE: Organic Letters (2001), 3(13), 2077-2079  
CODEN: ORLEF7; ISSN: 1523-7060  
PUBLISHER: American Chemical Society  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
OTHER SOURCE(S): CASREACT 135:137247  
REFERENCE COUNT: 25 THERE ARE 25 CITED REFERENCES AVAILABLE FOR  
THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE  
FORMAT

L12 ANSWER 17 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
AB 4-Nitrodiphenylamines (e.g., 4-nitrodiphenylamine) are prepared in high  
yield and selectivity by the amination of 4-nitrohalobenzenes (e.g.,  
4-chloronitrobenzene) with primary aromatic amines (e.g., aniline)  
in the presence of alkali carbonates (e.g., potassium carbonate) and  
copper-compound (e.g., copper oxide) catalysts  
with the addition of alkylsulfonyl halides (e.g., methanesulfonyl  
chloride)  
or arylsulfonyl halides to the reaction mixture  
ACCESSION NUMBER: 2001:150599 CAPLUS  
DOCUMENT NUMBER: 134:193204  
TITLE: Process and catalyst system for the  
preparation of 4-nitrodiphenylamines by the amination  
of 4-nitrohalobenzenes with primary aromatic amines  
in the presence of organosulfonyl halides and alkali  
carbonates  
INVENTOR(S): Schubart, Ruediger; Preuss, Reinhard  
PATENT ASSIGNEE(S): Bayer A.-G., Germany  
SOURCE: Ger. Offen., 4 pp.  
CODEN: GWXXBX  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 19940863	A1	20010301	DE 1999-19940863	19990827

PRIORITY APPLN. INFO.: DE 1999-19940863 19990827

OTHER SOURCE(S): CASREACT 134:193204; MARPAT 134:193204

L12 ANSWER 18 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB (meth)acrylamide and (meth)acrylate monomers containing oligoaniline side chain units were prepared by a modified Ullman condensation reaction to prepare the arylamine side groups with Cu as reactant and catalyst or by Pd catalyzed amination of aryl halides and triflates. The monomers prepared are N-(4-anilinophenyl)methacrylamide (M1), N-[4-(N'-acetyl-N'-phenyl)amino]phenyl methacrylamide (M2), and (M4); other monomers were also prepared by the method of D. Braun and S. Hauge (1971). Free radical polymerization using AIBN initiator of these monomers produces polymers with oligoanilines incorporated into the polymer as side chains with control of the side chain length and content of electroactive species. The solubility of the polymers is dependent on the extent of acetyl substitution, the inherent viscosity is 0.1 to 0.3 dL/g, and. The glass transition temperature of the homo-poly(methacrylamide)s is 183, 220, and 207°, for M1, M2, and M4, resp.

ACCESSION NUMBER: 2000:594182 CAPLUS  
 DOCUMENT NUMBER: 133:310209  
 TITLE: Synthesis and characterization of polymers with oligoaniline side chains  
 AUTHOR(S): Benicewicz, Brian C.; Chen, Ru  
 CORPORATE SOURCE: Department of Chemistry Rensselaer Polytechnic Institute, New York State Center for Polymer Synthesis, Troy, NY, 12180, USA  
 SOURCE: Polymer Preprints (American Chemical Society, Division of Polymer Chemistry) (2000), 41(2), 1733-1734  
 CODEN: ACPAPY; ISSN: 0032-3934  
 PUBLISHER: American Chemical Society, Division of Polymer Chemistry  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 REFERENCE COUNT: 15 THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS  
 RECORD. ALL CITATIONS AVAILABLE IN THE RE  
 FORMAT

L12 ANSWER 19 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Weak acid blue BS dye was prepared by treating bromamine acid with 4-dodecylaniline in the presence of soap emulsifier, CuSO4/SnCl2 catalyst, and NaHCO3, refining with glacial acetic acid, washing, drying, treating with fumed H2SO4, neutralizing, salting out, and drying.

ACCESSION NUMBER: 2000:467272 CAPLUS  
 DOCUMENT NUMBER: 133:60055  
 TITLE: Process improvement for preparation of Weak Acid Blue BS  
 AUTHOR(S): Yu, Guoce  
 CORPORATE SOURCE: Dandong Chemical Institute of Light Industry, Dandong, 118002, Peop. Rep. China  
 SOURCE: Fibre Huagong (2000), 17(3), 22-23, 33  
 CODEN: FTHUHF; ISSN: 1004-8960  
 PUBLISHER: Dandong Qinghuagong Yanjiuyuan  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Chinese

L12 ANSWER 20 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB R1NR2R3 (R1, R3 = C6-24 aryl; R2 = H, C6-24 aryl; R1, R2, and/or R3 contain electron-withdrawing group), which are useful as charge-transport agents for electroluminescent devices, are prepared by reaction of R1NR2 (R1, R2 = same as above) with R3X (R3 = same as above; X = halo) in the presence of compds. containing Cu or Cu salts. p-nitroaniline was reacted with p-iodonitrobenzene in the presence of K2CO3 and copper iodide in DMF at 200-205° for 2 h to give 93% 4,4'-dinitrophenylamine.

ACCESSION NUMBER: 2000:266253 CAPLUS  
 DOCUMENT NUMBER: 132:278974  
 TITLE: Preparation of aromatic amino compounds having electron-withdrawing group  
 INVENTOR(S): Kawamura, Haruyuki; Higaki, Kiyoshi  
 PATENT ASSIGNEE(S): Idemitsu Kosan Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokyo Koho, 12 pp.  
 CODEN: JKOXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2000119230	A2	20000425	JP 1998-290258	19981013
PRIORITY APPLN. INFO.:			JP 1998-290258	19981013

OTHER SOURCE(S): CASREACT 132:278974; MARPAT 132:278974

L12 ANSWER 21 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB This paper presents the results obtained from the oxidation of the aminoazobenzene dye AO52 by the UV/H2O2, UV/TiO2 and VIS/TiO2 systems.

In the former case, the authors investigated the formation of first byproducts by means of GC/MS, HPLC and 1HNMN. spectroscopy. The authors conclude that hydroxyl radicals are added to aromatic rings in the ipso position with respect to the sulfonate group or to the azo-linkage-bearing carbon. The reaction of the inorg. radical with the N,N-dimethylamino substituent, leading to demethylation, adds to the multiplicity of the possible pathways. Degradation by the UV/TiO2 system is pH dependent. Whereas hydroxyl radicals are the main oxidative agent in neutral and alkaline solns., pos. hole-induced oxidation competes with the reduction of the protonated dye mols. in acid media. Moreover, FTIR spectroscopy of AO52/TiO2 wafers provided an insight to the nature of the photoproducts. This process is very efficient since only ultimate breakdown products, i.e. aliphatic acids and inorg. salts, are detected. Similar results were obtained using visible light as the irradiation source in the case of wafers whereas in heterogeneous solns., the dye seems to be resistant to degradation

ACCESSION NUMBER: 1999:759222 CAPLUS  
 DOCUMENT NUMBER: 132:158798  
 TITLE: Photodegradation of the aminoazobenzene Acid orange 52 by three advanced oxidation processes: UV/H2O2, UV/TiO2 and VIS/TiO2 Comparative mechanistic and kinetic investigations  
 AUTHOR(S): Galindo, C.; Jacques, P.; Kalt, A.  
 CORPORATE SOURCE: Laboratoire de Chimie Textile, Ecole Nationale Supérieure de Chimie de Mulhouse, Mulhouse, F 68093, Fr.  
 SOURCE: Journal of Photochemistry and Photobiology, A: Chemistry (2000), 130(1), 35-47  
 CODEN: JPPCEJ; ISSN: 1010-6030  
 PUBLISHER: Elsevier Science S.A.  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 REFERENCE COUNT: 33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS  
 RECORD. ALL CITATIONS AVAILABLE IN THE RE  
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L12 ANSWER 22 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB The transition metal-catalyzed amination of aryl halides, in conjunction with an orthogonal protective group scheme, forms the basis of two routes to oligoaniline precursors. The oligoaniline precursors are soluble in a variety of common organic solvents, and are easily converted to the deprotected oligoanilines. The method allows the preparation of oligoanilines of even or odd chain lengths, and the incorporation of a variety of functional groups into the oligomers. Polyanilines of low polydispersity can also be prepared by this method.

ACCESSION NUMBER: 1999:375523 CAPLUS  
 DOCUMENT NUMBER: 131:19472  
 TITLE: Synthesis of oligoarylamines, and uses and reagents related thereto  
 INVENTOR(S): Singer, Robert A.; Sadighi, Joseph P.; Buchwald, Stephen L.; Mackewitz, Thomas  
 PATENT ASSIGNEE(S): Massachusetts Institute of Technology, USA  
 SOURCE: PCT Int. Appl., 142 pp.  
 CODEN: PIXXD2  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9928290	A1	19990610	WO 1998-US25555	19981202
W: CA, JP RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
US 6235871	B1	20010522	US 1998-203726	19981202
PRIORITY APPLN. INFO.:			US 1997-67275P	P 19971203

OTHER SOURCE(S): MARPAT 131:19472  
 REFERENCE COUNT: 6  
 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE  
 FORMAT

L12 ANSWER 23 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Diphenylamines 4-R1R2NC6H4N(COR3)C6H4-3-OR4 (R1, R3 = C1-8 alkyl; R2, R4 = H, C1-8 alkyl; R1 and R2 together may form a ring) are manufactured by reacting m-R3CONHC6H4OR4 and p-NR1R2C6H4X (X = halogen) in the presence of Cu compds. and tertiary amines. The amines are useful as precursors for fluoran dyes for thermal or pressure-sensitive recording materials. Thus, reacting m-N-acetylanisidine with 4-bromodimethylaniline in the presence of Cu(NO3)2 and 1,10-phenanthroline gave N-acetyl-4-(N',N'-dimethylamino)-3'-methoxydiphenylamine (m.p. 76-78°, yield 77.0%).

ACCESSION NUMBER: 1997:702027 CAPLUS  
 DOCUMENT NUMBER: 127:359972  
 TITLE: Diphenylamines for preparation of fluoran dyes and manufacture of the compounds  
 INVENTOR(S): Suga, Shigeki; Hidaka, Tomoya; Kawabe, Toru  
 PATENT ASSIGNEE(S): Nippon Soda Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.  
 CODEN: JKKXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 09278730	A2	19971028	JP 1996-110171	19960405
PRIORITY APPLN. INFO.:			JP 1996-110171	19960405

OTHER SOURCE(S): MARPAT 127:359972

L12 ANSWER 24 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Alkylarylamines are manufactured by reductive alkylation of aromatic amines with aliphatic ketones, whereby the mixture is subjected to the action of an acidic, heterogeneous catalyst (0.05-20% based on amine), at 20-200° for 0.1-20 h, and the obtained mixture including catalyst is hydrogenated in the presence of a Cu-based catalyst. The acidic heterogeneous catalysts include natural or synthetic amorphous aluminosilicates, natural or synthetic zeolites, HX or HY mol. sieves, aluminoborate or aluminochromate catalysts, zirconium phosphate, or bleaching clays or kieselguhr activated with mineral acids. For instance, a mixture of 460 g aniline, 573 g acetone, and 5 g amorphous aluminosilicate catalyst (75% SiO2, 25% Al2O3) in an autoclave was de-aerated and stirred for 2 h while raising the temperature to 150°, followed by hydrogenation at 5 MPa for 300 min. Conversion of aniline to N-isopropylaniline was 99%, and anal. of remaining low-boilers showed 76% acetone and 24% isopropanol.

ACCESSION NUMBER: 1997:331805 CAPLUS  
 DOCUMENT NUMBER: 126:305462  
 TITLE: Manufacturing process for alkyl aryl amines using reductive alkylation of aromatic amines.  
 INVENTOR(S): Volf, Jiri; Pasek, Josef; Mlynar, Jaromir; Dudek, Ivan; Seben, Stefan  
 PATENT ASSIGNEE(S): Vysoka Skola Chemicko Technologicka, Slovakia; VUCHT A. S.; Duslo A. S.  
 SOURCE: Slovakia, 3 pp.  
 CODEN: SLXXFO  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Slovak  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
SK 278291	B6	19960807	SK 1990-6167	19901211
PRIORITY APPLN. INFO.:			SK 1990-6167	19901211

OTHER SOURCE(S): CASREACT 126:305462

L12 ANSWER 25 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB In the catalytic reductive alkylation of aromatic amines by aliphatic ketones, using catalysts based on Cu and heterogeneous acid catalysts, selectivity is improved by: (1) starting with a mixture of the amine, ketone, and heterogeneous acid catalyst, (2) distilling off 3-98% of the stoichiometric water of reaction, (3) returning the used acid catalyst, and (4) hydrogenating the mixture. The method suppresses parallel reduction of the ketone to the corresponding alc.

For instance, in the alkylation of 280 g aniline by 600 g 4-methyl-2-pentanone, the reactants were refluxed with 4 g of an H-form amorphous aluminosilicate catalyst, with azeotropic distillation of 30 g H2O phase. The reaction product and the aluminosilicate catalyst were then autoclaved with 6 g Cu chromite catalyst at 180° and 5 MPa H2 pressure for 120 min, giving 99.9% conversion of aniline to N-(1,3-dimethylbutyl) aniline, and with recovered low-boiling substances showing 94% starting ketone and only 6% 4-methyl-2-pentanol. In contrast, a prior art process using only the Cu chromite catalyst gave only 65% conversion of aniline, with low-b. compds. showing only 15% recovered ketone and 85% 4-methyl-2-pentanol. The products are useful as antioxidants in the rubber industry.

ACCESSION NUMBER: 1997:244029 CAPLUS  
 DOCUMENT NUMBER: 126:225093  
 TITLE: Method of increasing of selectivity of catalytic reductive alkylation of aromatic amines with aliphatic ketones  
 INVENTOR(S): Volf, Jiri; Pasek, Josef; Mlynar, Jaromir; Dolezel, Pavel; Gruener, Alexander  
 PATENT ASSIGNEE(S): Vysoka Skola Chemicko Technologicka, Czech Rep.; Duslo  
 SOURCE: a.s.; VUCHT a.s. Slovakia, 3 pp.  
 CODEN: SLXXFO  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Slovak  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
SK 278068	B6	19951206	SK 1990-6404	19901219
PRIORITY APPLN. INFO.:			SK 1990-6404	19901219

OTHER SOURCE(S): CASREACT 126:225093

L12 ANSWER 26 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB The liquid phase formation of N-(alkyl)arylamines via the nickel-catalyzed alkylation of primary aryl amines with primary aliphatic alcs. was studied.  
 The nickel-catalyzed preparation of N-heptylaniline was compared against Pt, Pd, Ru, Rh, and Re/C as well as Co/k, Cu chromite, and PtSk/C. Several N,N'-dialkylphenylenediamines and N-alkyl-N'-phenylphenylenediamines were also prepared. The nickel-catalyzed reaction appeared to be a useful synthetic method for preparation of N-(alkyl)arylamines.

ACCESSION NUMBER: 1996:683485 CAPLUS  
 DOCUMENT NUMBER: 126:7754  
 TITLE: Preparation of N-(alkyl)arylamines. Nickel-catalyzed alkylation of primary aryl amines with primary aliphatic alcohols  
 AUTHOR(S): Reynolds, Michael P.; Greenfield, Harold  
 CORPORATE SOURCE: Uniroyal Chemical Co., Inc., Naugatuck, CT, 06770, USA  
 SOURCE: Chemical Industries (Dekker) (1996), 68(Catalysis of Organic Reactions), 343-351  
 CODEN: CHEIDI; ISSN: 0737-8025  
 PUBLISHER: Dekker  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English

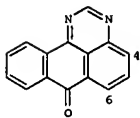
L12 ANSWER 27 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB The title dye is produced in improved yield from bromaminic acid and 3-(2-sulfoethylsulfonyl)aniline when using elemental Cu as catalyst in the presence of a mixture of NaH2PO4 and Na2HPO4 as buffer.

ACCESSION NUMBER: 1996:117938 CAPLUS  
 DOCUMENT NUMBER: 124:148721  
 TITLE: Preparation of C.I. Reactive Blue 19.  
 INVENTOR(S): von der Eltz, Andreas  
 PATENT ASSIGNEE(S): Hoechst A.-G., Germany  
 SOURCE: Ger. Offen., 5 pp.  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 4422160	A1	19960104	DE 1994-4422160	19940624
DE 4422160	C2	20030227		
CH 689425	A	19990415	CH 1995-1790	19950619
JP 08060015	A2	19960305	JP 1995-156459	19950622
CN 1127277	A	19960724	CN 1995-109172	19950622
CN 1072249	B	20011003		
BR 9502922	A	19960227	BR 1995-2922	19950623
PRIORITY APPLN. INFO.:			DE 1994-4422160	A 19940624

OTHER SOURCE(S): CASREACT 124:148721; MARPAT 124:148721

L12 ANSWER 28 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
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AB Pyrimidinoanthrone (I) reacts with N- and O-nucleophiles at positions 4 and/or 6, depending on the nucleophile, the catalyst, and the reaction medium.

ACCESSION NUMBER: 1995:373241 CAPLUS  
 DOCUMENT NUMBER: 122:265328  
 TITLE: Substitution of hydrogen atoms in pyrimidinoanthrone with N- and O-nucleophiles  
 AUTHOR(S): Kazankov, M. V.; Fotova, O. A.  
 CORPORATE SOURCE: Nauchno-Issled. Inst. Org. Polyprod. Krasitel., Moscow, Russia  
 SOURCE: Zhurnal Organicheskoi Khimii (1994), 30(6), 930-5  
 CODEN: ZORKAE; ISSN: 0514-7492  
 PUBLISHER: Nauka  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Russian

L12 ANSWER 29 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB The title compds. are manufactured by treating 1-amino-4-bromoanthraquinone-2-sulfonic acid (I) or salts with PhNH2 in an aqueous medium containing an acid binder and a Cu catalyst, heating in the presence of mineral acid, and separating 1-amino-4-anilinoanthraquinone-2-sulfonic acid (III). Treating 211.5 parts I Na salt with 182 parts PhNH2 in the presence of soda ash and CuSO4, heating at 90° in the presence of aqueous HCl, filtering, washing with aqueous NaCl, dispersing the wet cake in water, neutralizing with NaOH, and drying to give 230 parts II Na salt containing 80 mg Cu/kg.

ACCESSION NUMBER: 1995:118651 CAPLUS  
 DOCUMENT NUMBER: 122:83714  
 TITLE: Manufacture of anthraquinone compounds for acidic blue dyes  
 INVENTOR(S): Inoe, Masahito; Takahashi, Sho  
 PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 3 pp.  
 CODEN: JIQQAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 06192581	A2	19940712	JP 1992-344343	19921224
JP 3239496	B2	20011217		
PRIORITY APPLN. INFO.:			JP 1992-344343	19921224

L12 ANSWER 30 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Scale inhibitors are prepared by condensation of aromatic amines in the presence of catalysts selected from halogen-containing oxy acids or their salts, peroxides, azo compds., Fe or Cu oxides, halides, or sulfates, and aromatic nitro compds. and reaction inhibitors. Thus, a condensation product of 6-aminobenzenesulfonic acid with 4-aminodiphenylamine was coated on a reactor, which was used for suspension polymerization of vinyl chloride, showing scale deposition (after 100 batches) 0 and 18 g/m<sup>2</sup> in the liquid and liquid-gas interface portions of the reactor, resp.

ACCESSION NUMBER: 1994:580541 CAPLUS  
 DOCUMENT NUMBER: 121:180541  
 TITLE: Scale-preventing agents and coated reactors for polymerization of vinyl monomers  
 INVENTOR(S): Shimizu, Toshihide; Watanabe, Mikio  
 PATENT ASSIGNEE(S): Shinetsu Chemical Industry Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 22 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 2  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 06100604	A2	19940412	JP 1993-26248	19930121
US 5457170	A	19951010	US 1994-366056	19941229
PRIORITY APPLN. INFO.:			JP 1992-72910	A1 19920224
			JP 1992-233132	A1 19920807
			JP 1992-233133	A1 19920807
			JP 1991-302294	A 19911021
			JP 1991-359427	A 19911227
			JP 1991-359429	A 19911227
			JP 1991-359430	A 19911227
			JP 1992-72911	A 19920224
			JP 1992-177467	A 19920611
			US 1992-964267	A3 19921021

OTHER SOURCE(S): MARPAT 121:180541

L12 ANSWER 32 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB An efficient alternative to the copper-catalyzed synthesis (Ullmann ether synthesis) of biaryl ethers, biaryl thioethers, and biaryl amines involving the potassium fluoride-alumina-mediated addition of a phenol, thiophenol, or an aniline to 2- or 4-fluorobenzonitriles catalyzed by 18-crown-6 is described. An efficient alternative to the copper-catalyzed synthesis (Ullmann ether synthesis) of biaryl ethers, biaryl thioethers, and biaryl amines involving the potassium fluoride-alumina-mediated addition of a phenol, thiophenol, or an aniline to 2- or 4-fluorobenzonitriles catalyzed by 18-crown-6 is described.

ACCESSION NUMBER: 1993:472303 CAPLUS  
 DOCUMENT NUMBER: 119:72303  
 TITLE: Synthesis of diaryl ethers, diaryl thioethers, and diaryl amines mediated by potassium fluoride-alumina and 18-crown-6  
 AUTHOR(S): Schmittling, Elisabeth A.; Sawyer, J. Scott  
 CORPORATE SOURCE: Lilly Res. Lab., Eli Lilly and Co., Indianapolis, IN, 46385, USA  
 SOURCE: Journal of Organic Chemistry (1993), 58(12), 3229-30  
 CODEN: JOCEAH; ISSN: 0022-3263  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 119:72303

L12 ANSWER 31 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB A study of some parameters which influence the Ullmann-Goldberg condensation for the synthesis of N-phenylanthranilic acids was done, showing that these acids can be obtained efficiently using water as the solvent. Thus, 2-ClC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H was treated with RC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> (R = H, 4-Me, 3-O<sub>2</sub>N, 3-Cl, 4-H<sub>2</sub>N, 4-MeO, 4-HO<sub>2</sub>S) in refluxing H<sub>2</sub>O in the presence of powdered Cu to give 9-89% 2-(RC<sub>6</sub>H<sub>4</sub>NH)C<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H.

ACCESSION NUMBER: 1993:580482 CAPLUS  
 DOCUMENT NUMBER: 119:180482  
 TITLE: Synthesis of N-phenylanthranilic acid using water as solvent  
 AUTHOR(S): Pellon, Rolando F.; Carrasco, Ramon; Rodes, Lorenzo  
 CORPORATE SOURCE: Cent. Quim. Farm., Havana, Cuba  
 SOURCE: Synthetic Communications (1993), 23(10), 1447-53  
 CODEN: SYNGAV; ISSN: 0039-7911  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 119:180482

L12 ANSWER 33 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB PhNHCHMe<sub>2</sub> (I), a starting material for the herbicide Ramrod, is produced more economically by combining its production with that of the antiozonant p-(PhNH)C<sub>6</sub>H<sub>4</sub>NHCHMe<sub>2</sub> (II). Specifically, I and II are simultaneously produced by Cu-catalyzed reductive alkylation of a mixture of PhNH<sub>2</sub> (III) and 4-(PhNH)C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> (IV), containing 0.1-10% III, with Me<sub>2</sub>CO at 140-180° and 3-10 MPa. Unreacted Me<sub>2</sub>CO, alc., and reaction H<sub>2</sub>O are removed by fractional distillation, and vacuum distillation of the remainder gives a distilled concentrate containing I, and a residue containing II, with further distillation of the concentrate giving pure I. In the further (batch) distillation of the concentrate, the fraction containing III is returned to the alkylation, the fraction distilling with I but containing mostly PhNH<sub>2</sub> is removed, and the distillation residue is added to the post-reaction mixture. For example, to a continuous reactor was fed IV containing 0.3% III, and Me<sub>2</sub>CO (3 mol vs. amine), over a Cu chromite catalyst (concentration 0.5% vs. injected amine) at 150-170° and 5 MPa. Continuous distillation of the product with 3 columns gave in order: (1) Me<sub>2</sub>CO, Me<sub>2</sub>CHOH, and H<sub>2</sub>O, (2) I concentrate, and (3) pure IV. The concentrate containing 35% I, 40% II, 3% III, and 10% PhNH<sub>2</sub> was vacuum distilled as described, giving 3 kg pure I per ton II, with addnl. recovery of 4 kg II.

ACCESSION NUMBER: 1993:212652 CAPLUS  
 DOCUMENT NUMBER: 118:212652  
 TITLE: Improved method of producing N-isopropylaniline by combination with production of N-isopropyl-N'-phenyl-p-phenylenediamine  
 INVENTOR(S): Pasek, Josef; Mrazova, Celina; Jaros, Alois; Dolezel, Pavel; Halomi, Milan; Dudek, Ivan; Skrada, Dusan; Macak, Martin  
 PATENT ASSIGNEE(S): Czech., 4 pp.  
 SOURCE: CODEN: CZXXA9  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Czech  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 273842	B1	19910411	CS 1989-1902	19890328
PRIORITY APPLN. INFO.:			CS 1989-1902	19890328

OTHER SOURCE(S): CASREACT 118:212652

L12 ANSWER 34 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB p-Phenylenediamine (I), diphenylamine, phenyl-p-phenylenediamine, N,N'-diphenyl-p-phenylenediamine (II), and N,N'-diphenylbenzidine (III) are polymerized by Cu(ClO<sub>4</sub>)<sub>2</sub>·6H<sub>2</sub>O in acetonitrile or (NH<sub>4</sub>)<sub>2</sub>SO<sub>8</sub> in dilute acids. Elemental anal., thermogravimetric anal., IR and UV-Vis absorption, and XPS are used to characterize the polymers.

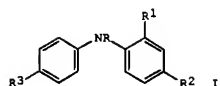
The polymers from I and II are most similar to polyaniline. The d.p. of poly-III is very low. In the as-synthesized salts, protonation may occur at either the imine or amine units. The polymers are susceptible to forming covalent bonds with Cl when HCl is used as the protonic acid during preparation

ACCESSION NUMBER: 1992:470399 CAPLUS  
 DOCUMENT NUMBER: 117:70399  
 TITLE: Structural investigations of aromatic amine polymers  
 AUTHOR(S): Neoh, K. G.; Kang, E. T.; Tan, K. L.  
 CORPORATE SOURCE: Dep. Chem. Eng., Nati. Univ. Singapore, 0511, Singapore  
 SOURCE: Journal of Physical Chemistry (1992), 96(16), 6777-83  
 CODEN: JPCHAX; ISSN: 0022-3654  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English

L12 ANSWER 35 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Aryllead triacetates are regioselective reagents for the mono-N-arylation of aromatic, heterocyclic, and aliphatic amines under mild and neutral conditions in a reaction catalyzed by copper diacetate. The arylation of arylamines was unaffected by the steric hindrance of the arylamine but was dependent on the arylamine basicity. The position of oxidizable substituents on both the aryllead triacetate and the arylamine was important due to a competing oxidation-reduction reaction. The arylation of heterocyclic amines proceeded in modest to good yields while aliphatic amines were arylated in poor to modest yields. The mechanism proposed for these reactions involves transfer of the aryl group onto copper forming a copper(III) intermediate which subsequently undergoes ligand coupling to give the N-arylated amine and the catalytic Cu (I) species.

ACCESSION NUMBER: 1991:655700 CAPLUS  
 DOCUMENT NUMBER: 115:255700  
 TITLE: Aryllead triacetates: regioselective reagents for N-arylation of amines  
 AUTHOR(S): Barton, Derek H. R.; Donnelly, Dervilla M. X.; Finet, Jean Pierre; Guiry, Patrick J.  
 CORPORATE SOURCE: Dep. Chem., Texas A and M Univ., College Station, TX, 77843, USA  
 SOURCE: Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999) (1991), (9), 2095-102  
 CODEN: JCPRB4; ISSN: 0300-922X  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 115:255700

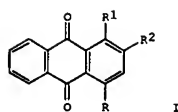
L12 ANSWER 36 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
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AB The byproducts in the tech.-grade N-nitrosodiphenylamine were identified as nitro and nitrosoderivs. (I; R = H, NO; R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub> = H, NO<sub>2</sub>).

ACCESSION NUMBER: 1991:121606 CAPLUS  
 DOCUMENT NUMBER: 114:121606  
 TITLE: Impurities in technical-grade. N-nitrosodiphenylamine  
 AUTHOR(S): Petrisko, Miroslav; Mrazova, Celina; Pasek, Josef  
 CORPORATE SOURCE: Vysk. Ustav Chem. Technol. S.P., Bratislava, Czech.  
 SOURCE: Chemicky Prumysl (1990), 40(7), 353-8  
 CODEN: CHPUA4; ISSN: 0009-2789  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Czech

L12 ANSWER 37 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
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AB Nucleophilic substitution reactions of bromoanthraquinones I [R = Br; (R<sub>1</sub>,R<sub>2</sub>) = (NH<sub>2</sub>,SO<sub>3</sub>Na) (II), (NH<sub>2</sub>,H) (III), (H,SO<sub>3</sub>Na) (IV), (H,H) (V)] with PhNH<sub>2</sub>, affording I [R=NHPh, OH, H; (R<sub>1</sub>,R<sub>2</sub>) same], were studied in the presence and absence of CuSO<sub>4</sub> catalyst. Product ratios depended on both catalyst and ambient oxygen. Substituent effects in the uncatalyzed reaction reflected their electron-accepting character, with the rate constant decreasing in the series IV > V > II > III; in the catalyzed reaction, the order was II > IV > III > V.

ACCESSION NUMBER: 1990:118010 CAPLUS  
 DOCUMENT NUMBER: 112:118010  
 TITLE: Kinetics of reaction of 1-bromoanthraquinone and its amino and sulfo derivatives with aniline  
 AUTHOR(S): Vinokurov, Yu. V.; Nazarova, N. E.; Solodova, K. V.; Shein, S. M.  
 CORPORATE SOURCE: USSR  
 SOURCE: Zhurnal Organicheskoi Khimii (1989), 25(9), 1926-31  
 CODEN: ZORKAE; ISSN: 0514-7492  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Russian  
 OTHER SOURCE(S): CASREACT 112:118010

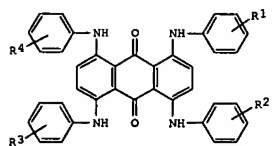
L12 ANSWER 38 OF 86 CAPLUS COPYRIGHT 2005 ACS ON STN  
 AB The condensation of 1-amino-4-bromoanthraquinone-2-sulfonic acid (I) with primary and secondary aliphatic and aromatic amines, diamines, and polyamines to give the title dyes and dye intermediates is efficiently catalyzed with Cu catalysts in the presence of pH regulators. The catalysts contain 36-85% Cu, Cu compds., or their mixts. deposited on a porous silicate carrier and may be prepared from catalysts recycled from hydrogenation and dehydrogenation reactions. A mixture of 31% aqueous MeNH<sub>2</sub> 20, I 18, powdered catalyst containing Cu and CuO on a porous silicate carrier 1, and Na<sub>2</sub>CO<sub>3</sub> 5.4 g was heated in 400 mL H<sub>2</sub>O to 80° for 2 h giving 84% 1-amino-4-(methylamino)-2-anthraquinonesulfonic acid.

ACCESSION NUMBER: 1990:38120 CAPLUS  
 DOCUMENT NUMBER: 112:38120  
 TITLE: Preparation of 1-amino-4-(aryl- or alkylamino)-2-anthraquinonesulfonic acids and their salts  
 INVENTOR(S): Horyna, Jaroslav; Slosar, Petr; Popova, Eva; Znamenacek, Milan  
 PATENT ASSIGNEE(S): Czech.  
 SOURCE: Czech., 8 pp. Addn. to Czech. 249,378.  
 CODEN: CZXXA9  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Czech  
 FAMILY ACC. NUM. COUNT: 2  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 256876	B3	19880415	CS 1985-4930	19850701
CS 249378	B1	19870312	CS 1985-870	19850208
			CS 1985-870	19850208

PRIORITY APPLN. INFO.:  
 OTHER SOURCE(S): CASREACT 112:38120

L12 ANSWER 39 OF 86 CAPLUS COPYRIGHT 2005 ACS ON STN  
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AB The title dyes I (R1-R4 = H, halogen, lower alkyl, cycloalkyl, lower alkoxy, CF<sub>3</sub>, PhO, OH; such that 21 of R1-R4 is halogen), which have very high near-IR absorption and are thus useful as organic filters for semiconductor laser-containing measuring apparatus, are prepared by reacting 1,4,5,8-tetrachloroanthraquinone (II) with a 24-fold molar excess of an appropriately substituted PhNH<sub>2</sub> in the presence of a catalytically effective amount of Cu ions, a salt of an aliphatic carboxylic acid (e.g., KOAc), and PhCH<sub>2</sub>OH or its derivs. at elevated temps. In this manner, II 10.87, p-toluidine 27.2, 4-ClC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> 31.3, KOAc 13.4, CuSO<sub>4</sub> 1.24, and PhCH<sub>2</sub>OH 3.41 parts were heated to 130° under N for 6.5 h, forming 1-(4-chloroanilino)-4,5,8-tris(4-methylanilino)anthraquinone, which had 99% transmittance (CHCl<sub>3</sub>) at 860 nm.

ACCESSION NUMBER: 1990:8710 CAPLUS  
 DOCUMENT NUMBER: 112:8710  
 TITLE: Halogenated anthraquinones useful as near infrared-absorbing dyes and their preparation  
 INVENTOR(S): Ohyamata, Tsukasa; Takuma, Keisuke; Kuroda, Shizuo; Aiga, Hiroshi  
 PATENT ASSIGNEE(S): Mitsui Toatsu Chemicals, Inc., Japan  
 SOURCE: Eur. Pat. Appl., 7 pp.  
 CODEN: EPXXDW  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 323184	A1	19890705	EP 1988-312285	19881223
EP 323184	B1	19940309		
R: CH, DE, FR, GB, LI, NL				
JP 01172458	A2	19890707	JP 1987-330163	19871228
JP 08013930	B4	19960214		
CA 1321790	A1	19930831	CA 1988-586468	19881220
US 5342974	A	19940830	US 1988-291028	19881228
PRIORITY APPLN. INFO.:			JP 1987-330163	A 19871228

OTHER SOURCE(S): MARPAT 112:8710

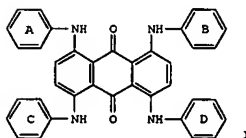
L12 ANSWER 40 OF 86 CAPLUS COPYRIGHT 2005 ACS ON STN  
 AB Title compds. p-PhNC<sub>6</sub>H<sub>4</sub>NHR (I; R = CHMe<sub>2</sub>, CHMeCH<sub>2</sub>CHMe<sub>2</sub>) were prepared by catalyzed reductive alkylation of p-PhNC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> (II) with Me<sub>2</sub>CO and MeCOCH<sub>2</sub>CHMe<sub>2</sub> and factors improving selectivity were studied. Thus, 1600 kg/h mixture containing Me<sub>2</sub>CO and II in molar ratio 3:1 and 0.3% CuCr catalyst (based on II) was passed consecutively through 3 reactors which were heated at 140°, 150°, and 160°, resp., and the contents were stirred with circulating H at a feed 1450 m<sup>3</sup>/h, 5.5 MPa, and stream velocity 0.4 m/s. The product discharged from the 3rd reactor was filtered and volatile components containing Me<sub>2</sub>CO:Me<sub>2</sub>CHOH in ratio 7:3 were separated to give I (R = CHMe<sub>2</sub>) containing 0.5% II and 1% diisopropyl derivs.

ACCESSION NUMBER: 1989:514837 CAPLUS  
 DOCUMENT NUMBER: 111:114837  
 TITLE: Process for continuous production of N-(secondary alkyl)-N'-phenyl-p-phenylenediamines  
 INVENTOR(S): Pasek, Josef; Waradzin, Walter; Dolezel, Pavel; Mosny,  
 Ivan; Uhlar, Jan; Masek, Jan; Mlynar, Jaromir; Volf, Jiri; Pexidr, Vaclav  
 PATENT ASSIGNEE(S): Czech.  
 SOURCE: Czech., 5 pp.  
 CODEN: CZXXA9  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Czech  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 254739	B1	19880115	CS 1986-5971	19860813
			CS 1986-5971	19860813

PRIORITY APPLN. INFO.:  
 OTHER SOURCE(S): CASREACT 111:114837

L12 ANSWER 41 OF 86 CAPLUS COPYRIGHT 2005 ACS ON STN  
 GI



AB Title materials I (rings A-D may be substituted with lower alkyl, cycloalkyl, lower alkoxy, CF<sub>3</sub>, OPh, OH, or halo), useful for near-IR filters, sunglasses, and optical recording materials, are prepared from 1,4,5,8-tetrachloroanthraquinone (II) and 24 mol aniline derivs. in the presence of CuSO<sub>4</sub>, (substituted) benzyl alc., and aliphatic carboxylate salts. Thus, 10.87 parts II and 54.35 parts p-toluidine were treated in the presence of AcOK, CuSO<sub>4</sub>, and benzyl alc. at 130° for 6.5 h to give a title coloring material.

ACCESSION NUMBER: 1989:424962 CAPLUS  
 DOCUMENT NUMBER: 111:24962  
 TITLE: Manufacture of greenish anthraquinone coloring materials  
 INVENTOR(S): Takuma, Hirotsuke; Karasawa, Akio; Kuroda, Shizuo; Aiga, Hiroshi  
 PATENT ASSIGNEE(S): Mitsui Toatsu Chemicals, Inc., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.  
 CODEN: JKOXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 63312362	A2	19881220	JP 1987-146872	19870615
JP 07017848	B4	19950301		
PRIORITY APPLN. INFO.:			JP 1987-146872	19870615

OTHER SOURCE(S): MARPAT 111:24962



L12 ANSWER 42 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB The title polymers are prepared by partially or fully polymerizing aniline compds. in the presence of cupric compds. and nitrile compds., adding pyrrole compds. to the mixture, and polymerizing the pyrrole compds. Thus, 2.8 g aniline and 150 mL acetonitrile (I) were stirred at 15-20 °C as 63.2 g aqueous 45% Cu(BF<sub>4</sub>)<sub>2</sub> and 75 mL I were added dropwise for 15 min to form a slurry, which was stirred 2 h and kept 12 h at room temperature. Then, 0.67 g pyrrole was added over 5 min, and the mixture was stirred 2 h and kept 12 h at room temperature to give 4.0 g powdered black polymer with elec. conductivity 1.4 × 10<sup>-2</sup> S cm<sup>-1</sup>.  
 ACCESSION NUMBER: 1989:194286 CAPLUS  
 DOCUMENT NUMBER: 110:194286  
 TITLE: Manufacture of conductive polymers for semiconductors  
 INVENTOR(S): Suzuki, Tetsuyoshi; Hasegawa, Kazumi; Ando, Osamu  
 PATENT ASSIGNEE(S): Mitsubishi Kasei Corp., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 10 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 63243161	A2	19881011	JP 1987-78109	19870331

PRIORITY APPLN. INFO.: JP 1987-78109 19870331

L12 ANSWER 43 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Title compds. with C2-12 alkyls are produced by alkylation of anilines with primary and secondary alcs. in the liquid phase in the presence of a Cu catalyst at 150-250°, H partial pressure 0.2-10 MPa, and an alc.-NH<sub>2</sub> group mol ratio of (0.5-5):1. The products are suitable for manufacture of dyes, pesticides, stabilizers for rubber and gunpowder, and rubber vulcanization accelerators (no data). Thus, 48 g 2:1 (mol ratio) EtOH-aniline mixture and 5 g 33:6 (I) Cu-MgO/SiO<sub>2</sub> catalyst were heated to 190° and 3-4.2 MPa H to give a mixture containing unreacted EtOH 53.0, unreacted aniline 11.9, Et<sub>3</sub>N 0.54, N-ethylaniline 30.65, N,N-diethylaniline 2.95, and phenylcyclohexylamine 0.06%.  
 ACCESSION NUMBER: 1988:630511 CAPLUS  
 DOCUMENT NUMBER: 109:230511  
 TITLE: Preparation of N-alkyl- and N,N-dialkylanilines  
 INVENTOR(S): Pasek, Josef; Dlouhy, Jiri; Rozehnal, Jiri; Volf, Jiri; Pavlas, Jiri  
 PATENT ASSIGNEE(S): Czech.  
 SOURCE: Czech., 9 pp.  
 CODEN: CZXXA9  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Czech  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 237663	B1	19850917	CS 1982-9867	19821228

PRIORITY APPLN. INFO.: CS 1982-9867 19821228

L12 ANSWER 44 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB The selectivity of Cu catalysts was increased by treatment with an organic acid for the title reaction to give RNHCHR1R2 [R = Ph, (substituted) aryl; R1, R2 = C1-4 alkyl]. Thus, heating a mixture of 25 g 4-PhNHC6H4NH<sub>2</sub>, 30 mL Me<sub>2</sub>CO, and 0.5 g com. Cu-Cr catalyst containing 1.65% AcOH for 80 min at 160° and H pressure 6 MPa in an autoclave gave a 99.49:0.51 mixture of 4-PhNHC6H4NHCHMe<sub>2</sub> and 4-PhNHC6H4N:CM<sub>2</sub> and 13.9% conversion of Me<sub>2</sub>CO to Me<sub>2</sub>CHOH.  
 ACCESSION NUMBER: 1988:589983 CAPLUS  
 DOCUMENT NUMBER: 109:189983  
 TITLE: Reductive alkylation of aromatic amines with ketones over copper catalysts  
 INVENTOR(S): Pasek, Josef; Jarkovsky, Lubor; Uhlar, Jan  
 PATENT ASSIGNEE(S): Czech.  
 SOURCE: Czech., 8 pp.  
 CODEN: CZXXA9  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Czech  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 235833	B1	19850515	CS 1983-2229	19830330

PRIORITY APPLN. INFO.: CS 1983-2229 19830330

OTHER SOURCE(S): CASREACT 109:189983

L12 ANSWER 45 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB The amines RC<sub>6</sub>H<sub>4</sub>NHC6H4NO<sub>2</sub> (R = H, Me, NO<sub>2</sub>, MeO, EtO) are prepared by condensing o- or p-halonitrobenzenes with RC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> in the presence of Cu complexes with N,N- or N,O-bidentate ligands and acid acceptors. Heating p-ClC<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>, PhNH<sub>2</sub>, K<sub>2</sub>CO<sub>3</sub> and bis(1,10-phenanthroline) copper(I) nitrate in PhMe at 180-90° for 8 h gave 34.3% p-nitrodiphenylamine.  
 ACCESSION NUMBER: 1988:572537 CAPLUS  
 DOCUMENT NUMBER: 109:172537  
 TITLE: Manufacture of nitrodiphenylamines in high yields  
 INVENTOR(S): Yoshimura, Masakatsu; Nuno, Tatsumi; Kurimoto, Isao  
 PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 63072658	A2	19880402	JP 1986-218758	19860917

PRIORITY APPLN. INFO.: JP 1986-218758 19860917

OTHER SOURCE(S): MARPAT 109:172537

L12 ANSWER 46 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB RC6H4NHC6H4NO2 (R = H, Me, NO2, OMe, OEt), useful as intermediates for dyes and agents for organic rubber processing, were prepared by treating XC6H4NO2 (X = o- or p-halo) with RC6H4NH2 in the presence of tetraalkylphosphonium salts, Na2CO3, and Cu catalysts. Thus, a mixture of p-ClC6H4NO2, PhNH2, Na2CO3, Cu oxide, and Bu4P+ Cl- was stirred at 180-190° under 500-750 mmHg for 10 h to give 86.9% p-PhNHC6H4NO2. The reaction did not proceed without Bu4P+ Cl-.

ACCESSION NUMBER: 1988:570014 CAPLUS  
 DOCUMENT NUMBER: 109:170014  
 TITLE: Catalytic preparation of nitrodiphenylamines from halonitrobenzenes and anilines  
 INVENTOR(S): Yoshimura, Masakatsu; Nuno, Tatsumi; Ebina, Chineto; Yamada, Akira  
 PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 3 pp.  
 CODEN: JKKXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 62283953	A2	19871209	JP 1986-128916	19860602

PRIORITY APPLN. INFO.: JP 1986-128916 19860602

OTHER SOURCE(S): CASREACT 109:170014

L12 ANSWER 47 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB N-Alkylamines are prepared by reaction of aromatic, alicyclic or heterocyclic amines with aliphatic alcs. (as alkylating agents) in the presence of Cu-Zn/Al2O3 catalyst in a fixed-bed reactor at 150-300°/1-10 kg/cm2. The catalyst contains 10-50% Cu and 10-70% Zn. Thus, a 1:2.24 (mol. ratio) PhNH2-MeOH mixture was passed over the catalyst at 250°, 1 kg/cm2, and 0.4 h-1 liquid space velocity to give 94.9% yield N-methylaniline at 99% once-through PhNH2 conversion.

ACCESSION NUMBER: 1988:133821 CAPLUS  
 DOCUMENT NUMBER: 108:133821  
 TITLE: Preparation of N-alkylamines  
 INVENTOR(S): Xu, Bingsheng; Ding, Li; Sun, Yan; Lou, Zhenhua; Wang, Weimin  
 PATENT ASSIGNEE(S): Fushun Petrochemical Research Institute, Sinopec, Peop. Rep. China  
 SOURCE: Faming Zhuanli Shenqing Gongkai Shuomingshu, 6 pp.  
 CODEN: CNXQEV  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Chinese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CN 85100326	A	19860917	CN 1985-100326	19850401
CN 85100326	B	19871202		

PRIORITY APPLN. INFO.: CN 1985-100326 19850401

L12 ANSWER 48 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Ph3Bi N-phenylates aliphatic and aromatic amines (e.g., PhNH2, Ph2NNH2, BuNH2, p-NO2C6H4NH2) in the presence of Cu(OAc)2.

ACCESSION NUMBER: 1988:21408 CAPLUS  
 DOCUMENT NUMBER: 108:21408  
 TITLE: Copper salt catalysis of N-phenylation of amines by trivalent organobismuth compounds  
 AUTHOR(S): Barton, Derek H. R.; Finet, Jean Pierre; Khamsi, Jamal  
 CORPORATE SOURCE: Inst. Chim. Subst. Nat., CNRS, Gif-sur-Yvette, 91190, Fr.  
 SOURCE: Tetrahedron Letters (1987), 28(8), 887-90  
 CODEN: TELEAY; ISSN: 0040-4039  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 108:21408

L12 ANSWER 49 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB The N-arylation of RR1NH (R = Bu, Et, cyclohexyl), PhCH2CHCO2Et, Ph2C:N, R1 = H, Et) and R2NH2 (R2 = Ph, 4-MeC6H4, 4-MeOC6H4, 2-, 4-O2NC6H4, mesityl) by Ph3BiR3R4 (R3, R4 = OAc, O2CCF3, 4-MeC6H4SO3, Ph; R3R4 = CO3) under neutral conditions is strongly catalyzed by Cu powder at room temperature to give PhR5R6N (R5, R6 = H, R, R1, R2).

ACCESSION NUMBER: 1987:406849 CAPLUS  
 DOCUMENT NUMBER: 107:6849  
 TITLE: Metallic copper catalysis of N-arylation of amines by triarylbiomuth diacylates  
 AUTHOR(S): Barton, Derek H. R.; Finet, Jean Pierre; Khamsi, Jamal  
 CORPORATE SOURCE: Inst. Chim. Subst. Nat., Gif-sur-Yvette, 91190, Fr.  
 SOURCE: Tetrahedron Letters (1986), 27(31), 3615-18  
 CODEN: TELEAY; ISSN: 0040-4039  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 107:6849

L12 ANSWER 50 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN

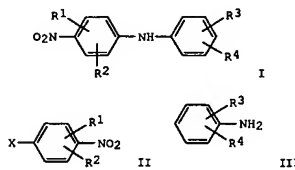
AB Elec. conductivity polyaniline is prepared by polymerization of PhNHC6H4NHPh (I) in the presence of chemical oxidants. NH4 persulfate (34.2 parts) dissolved in 200 parts H2O was added dropwise at 5° over 1 h to a mixture of H2O 1200, concentrated HCl 300, and p-I 13.0 parts, the mixture stirred at 5° for 6 h and at room temperature for 18 h forming polyaniline, which was press molded at 3800 kg/cm2 to show elec. conductivity 4.0 S/cm.

ACCESSION NUMBER: 1987:214630 CAPLUS  
DOCUMENT NUMBER: 106:214630  
TITLE: Polyaniline  
INVENTOR(S): Hagiwara, Tsuneo; Iwata, Kaoru  
PATENT ASSIGNEE(S): Agency of Industrial Sciences and Technology, Japan  
SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.  
CODEN: JKXXAF  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 61266434	A2	19861126	JP 1985-108283	19850522
JP 03046007	B4	19910712	JP 1985-108283	19850522

PRIORITY APPLN. INFO.:

L12 ANSWER 51 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
GI



AB The title compds. (I; R1-4 = H, alkyl), useful as stabilizers for rubber and as intermediates for dyes (no data), are prepared by condensation of nitrobenzenes II (X = Cl, Br) with anilines III in the presence of a Cu compound catalyst and an amide or nitrile cocatalyst. Thus, a mixture containing II (X = Cl, R1 = R2 = H) 157.6, PhNH2

186, K2CO3 100, CuO 2, and PhCONH2 3 g with 20 mL xylene was heated at 195° while a further 186 g PhNH2 was added to give 88% I (R1-4 = H). The entire process required 12 h.

ACCESSION NUMBER: 1987:101862 CAPLUS  
DOCUMENT NUMBER: 106:101862  
TITLE: N-phenyl-4-nitroanilines  
INVENTOR(S): Podder, Chiraranjan; Schlesmann, Hasso  
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.  
SOURCE: Ger. Offen., 14 pp.  
CODEN: GWXXBX  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

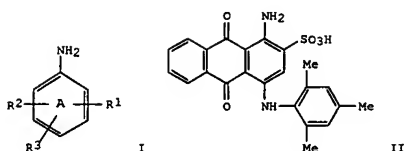
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3518272	A1	19861127	DE 1985-3518272	19850522
IN 167509	A	19901110	IN 1986-DE344	19860417
JP 61271257	A2	19861201	JP 1986-112825	19860519
US 4771067	A	19880913	US 1987-90430	19870826

PRIORITY APPLN. INFO.:

US 1986-862608	A1	19860512
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OTHER SOURCE(S): CASREACT 106:101862

L12 ANSWER 52 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
GI



AB Anthraquinone dyes are prepared by the condensation in aqueous medium of 1-amino-4-bromoanthraquinone-2-sulfonic acid with water-insol. amines (I; R1-R3 = H, alkyl, alkoxy; ring A may be benzene or cyclohexane) in the presence of 10-25 volume% nonionic surfactant at 65-100°, with Cu or Cu salt catalysts. Thus, 1-amino-4-bromoanthraquinone-2-sulfonic acid was mixed with 2,4,6-trimethylaniline, polyethylene glycol, Fraewozell N 55, and water and heated to 40° with intensive stirring. NaHCO3 was added and the reaction mixture heated to 65-75°, at which temperature CuCl was added,

and the mixture refluxed for 6-8 h. By acidification the product was precipitated, filtered, and dried, forming II as blue-violet crystals.

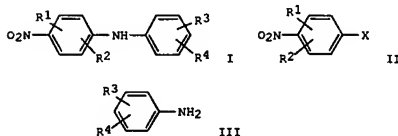
ACCESSION NUMBER: 1986:610411 CAPLUS  
DOCUMENT NUMBER: 105:210411  
TITLE: Anthraquinone dyes  
INVENTOR(S): Shlykov, Yu.; Hepp, Wulfdieter; Knoechel, Gerhard; Schick, Erhard; Riedel, Hans Juergen; Benndorf, Ulrich; Weise, Heinz  
PATENT ASSIGNEE(S): VEB Chemiekombinat Bitterfeld, Ger. Dem. Rep.  
SOURCE: Ger. (East), 6 pp.  
CODEN: GEXXAB  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DD 226294	A1	19850821	DD 1984-265289	19840716

PRIORITY APPLN. INFO.:

DD 1984-265289	19840716
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L12 ANSWER 53 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
GI



AB 4-Nitrodiphenylamines (I; R1-R4 = H, Cl-9 alkyl) are prepared by condensation of halonitrobenzenes II (X = Br, Cl) with anilines III in the presence of a Cu compound, K2CO3, and a polyamide. Thus, a mixture of 157.6 g II (R1 = R2 = H, X = Cl), 186 g PhNH2, 100 g K2CO3, 20 mL xylene, 2 g Cu oxide and 2.5 g polyamide 6 was heated at 195° to give 88.4% I (R1-R4 = H).

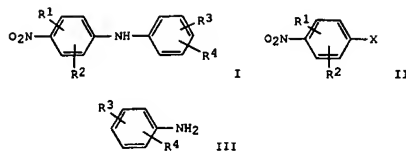
ACCESSION NUMBER: 1986:590649 CAPLUS  
DOCUMENT NUMBER: 105:190649  
TITLE: 4-Nitrodiphenylamines  
INVENTOR(S): Podder, Chiraranjan; Schlesmann, Harro  
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.  
SOURCE: Ger. Offen., 14 pp.  
CODEN: GWXXBX  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3504479	A1	19860814	DE 1985-3504479	19850209
IN 166356	A	19900414	IN 1986-DE24	19860108
US 4670595	A	19870602	US 1986-825216	19860203
JP 61183251	A2	19860815	JP 1986-21336	19860204

PRIORITY APPLN. INFO.:

DE 1985-3504479	A	19850209
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OTHER SOURCE(S): CASREACT 105:190649



AB The nitrodiphenylamines I (R1,R2,R3,R4 = H, Cl-9 alkyl) are prepared by condensation of the halonitrobenzenes II (X = Cl, Br) with the anilines III, in the presence of K2CO3, a Cu compound, and a carbonic acid amide (urethane, urea, biuret, etc.). The II/III ratio should be 1:3-5. Thus, a mixture of 157.6 g II (R1 = R2 = H, X = Cl),

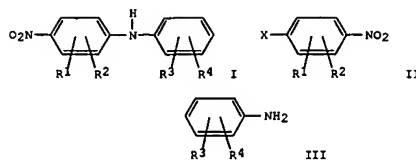
186 g PhNH2, 100 g K2CO3, 20 mL xylene, 2 g Cu oxide, and 2.23 g urethane was treated portion-wise with 186 g aniline at 195° to give 90.9% I (R1 = R2 = R3 = R4 = H). I can be reduced to aminodiphenylamines, which are dye intermediates.

ACCESSION NUMBER: 1986:572038 CAPLUS  
DOCUMENT NUMBER: 105:172038  
TITLE: Nitrodiphenylamines  
INVENTOR(S): Podder, Chiraranjan; Schlesmann, Harro  
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.  
SOURCE: Ger. Offen., 14 pp.  
CODEN: GWXXBX

DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3501698	A1	19860724	DE 1985-3501698	19850119
IN 165808	A	19900113	IN 1985-DE1072	19851217
US 4665232	A	19870512	US 1986-817502	19860109
JP 61167649	A2	19860729	JP 1986-5155	19860116
PRIORITY APPLN. INFO.:			DE 1985-3501698	A 19850119

OTHER SOURCE(S): CASREACT 105:172038



AB The title compds. I (R1-4 = H, Cl-9 alkyl), useful as intermediates for dyes and rubber stabilizers (no data), are prepared by substitution of halonitrobenzenes II (X = halo) with anilines III at 140-225° in the presence of a Cu catalyst and a proton acceptor. Thus, 157.6 g II (R1 = R2 = H, X = Cl) and 372 g III

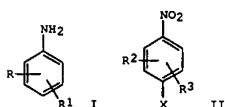
(R3 = R4 = H) in 120 g sulfolane and 20 mL xylene were heated at 195° in the presence of 2 g Cu oxide and 100 g K2CO3 to give 89% I (R1-4 = H).

ACCESSION NUMBER: 1986:514713 CAPLUS  
DOCUMENT NUMBER: 105:114713  
TITLE: 4-Nitrodiphenylamines  
INVENTOR(S): Podder, Chiraranjan; Schlesmann, Harro  
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.  
SOURCE: Ger. Offen., 15 pp.  
CODEN: GWXXBX

DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3443679	A1	19860605	DE 1984-3443679	19841130
IN 166353	A	19900414	IN 1985-DE894	19851025
US 4665233	A	19870512	US 1985-798906	19851118
JP 61134355	A2	19860621	JP 1985-265158	19851127
PRIORITY APPLN. INFO.:			DE 1984-3443679	A 19841130

OTHER SOURCE(S): CASREACT 105:114713



AB The N-arylation of anilines I (R and R1 are H, alkyl, alkoxy, cycloalkyl) by halobenzenes II (X = Cl, Br; R2 and R3 are H, alkyl) was catalyzed by Cu-Zn compound and Cu compound-Zn compound mixts. Thus, PhNH2 was treated with 4-ClC6H4NO2, CuO, and ZnO at 185-90° to give 4-O2NC6H4NHPh.

ACCESSION NUMBER: 1984:191557 CAPLUS  
DOCUMENT NUMBER: 100:191557  
TITLE: p-Nitrodiphenylamine  
INVENTOR(S): Sturm, Budd H.  
PATENT ASSIGNEE(S): Goodyear Tire and Rubber Co., USA  
SOURCE: U.S., 7 pp.  
CODEN: USXXAM

DOCUMENT TYPE: Patent  
LANGUAGE: English  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4435599	A	19840306	US 1982-444633	19821126
BR 8306216	A	19840731	BR 1983-6216	19831111
CA 1213615	A1	19861104	CA 1983-441077	19831114
EP 110810	A1	19840613	EP 1983-630192	19831118
EP 110810	B1	19860226		
R: BE, DE, FR, GB, IT, NL				
JP 5910640	A2	19840620	JP 1983-220487	19831122
JP 01049254	B4	19891024		
PRIORITY APPLN. INFO.:			US 1982-444633	A 19821126

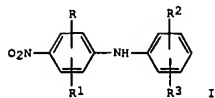
AB 1-Amino-4-bromo-2-anthraquinonesulfonic acid (I) [116-81-4] gives blue and green dyes when condensed with substituted anilines or cyclohexylamine [108-91-8] in the presence of 0.012-0.14 part soluble Cu(II) salt and 0.01-0.11 part SnCl2 and/or alkaline sulfite or metabisulfite per 1 parts I at pH 8-13 [in the presence of K Na tartrate (II)]. The condensation is faster than with other catalysts and side reactions are suppressed. Thus, I Na salt [6258-06-6] 7.4, 4-AcNHCH6H4NH2 [122-80-5] 3.5, NaHCO3 4.5, Na2CO3 0.9, H2O 100, and II 5 parts were heated to 70-80° with CuSO4.5H2O 0.9, SnCl2 0.8, and H2O 8 parts for 1 h to give 8.47 parts of crude dye and 7.9 parts pure dye { 6424-85-7} after chromatog. refining.

ACCESSION NUMBER: 1983:489626 CAPLUS  
DOCUMENT NUMBER: 99:89626  
TITLE: Condensation of bromamine acid with aromatic amines  
INVENTOR(S): Adamek, Milan; Kulic, Jiri; Poskocil, Jaroslav; Lustig, Jiri  
PATENT ASSIGNEE(S): Czech.  
SOURCE: Czech., 6 pp.  
CODEN: CZXXA9

DOCUMENT TYPE: Patent  
LANGUAGE: Czech  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 189516	B	19790430	CS 1977-6750	19771018
PRIORITY APPLN. INFO.:			CS 1977-6750	A 19771018

L12 ANSWER 58 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
GI



AB Nitrodiphenylamines I (R-R3 = H, alkyl) were prepared by condensation of  
a

halonitrobenzene with an aniline in presence of a Cu compound and a diaza heterocycle. Thus, aniline 93, CuO 2, and 1,8-diazabicyclo[5.4.0]undec-7-ene 3.8 g in 40 mL xylene were heated 20 min at 150°, treated with 4-ClC6H4NO2 157.7 and K2CO3 100 g, and heated 7 h at 193-196° to give 92.6% I (R-R3 = H). I are intermediates for dyes and rubber stabilizers.

ACCESSION NUMBER: 1983:405333 CAPLUS  
DOCUMENT NUMBER: 99:5333  
TITLE: 4-Nitrodiphenylamines  
INVENTOR(S): Heise, Klaus Peter; Wedemeyer, Karlfried  
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.  
SOURCE: Ger. Offen., 32 pp.  
CODEN: GWXXBX  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3137041	A1	19830324	DE 1981-3137041	19810917
US 4404400	A	19830913	US 1982-414224	19820902
EP 75174	A1	19830330	EP 1982-108191	19820906
EP 75174	B1	19850807		
R: BE, DE, FR, GB, IT, NL				
JP 58062140	A2	19830413	JP 1982-159017	19820914
JP 02026621	B4	19900612		

PRIORITY APPLN. INFO.: DE 1981-3137041 A 19810917

L12 ANSWER 59 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
AB A highly sensitive and selective catalytic method for the determination  
of

Cu(II) is proposed, based on the color reaction of N-phenyl-p-phenylenediamine [101-54-2] with N,N-dimethylaniline [121-69-7] in the presence of H2O2. Cu (II) concns. ≥10-9M are determined from the increase in the absorbance of the colored product at 728 nm at a fixed time after the initiation of the reaction. The method is useful for the anal. of Cu in tap and river waters, and there are few interferences.

ACCESSION NUMBER: 1983:166652 CAPLUS  
DOCUMENT NUMBER: 98:166652  
TITLE: Catalytic determination of nanogram amounts of copper(II) by the oxidative coupling reaction of N-phenyl-p-phenylenediamine with N,N-dimethylaniline  
AUTHOR(S): Nakano, Shigenori; Tanaka, Masaya; Fushihara, Masanori; Kawashima, Takuji  
CORPORATE SOURCE: Fac. Educ., Tottori Univ., Tottori, Japan  
SOURCE: Mikrochimica Acta (1983), 1(5-6), 457-65  
CODEN: MIACAQ; ISSN: 0026-3672  
DOCUMENT TYPE: Journal  
LANGUAGE: English

L12 ANSWER 60 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
AB Urethanes were prepared by treatment of primary amines with CO and a hydroxy compound in the presence of at least one noble metal or compound from Group VIII, and a quinoid compound. Thus, 487 g mixture containing 41 ppm Pd(OAc)2, 206 ppm Cu(OAc)2.2H2O 1.7 tetrachloro-p-benzoquinone, 0.8 PhNEt2, 89 EtOH, and 8.4 weight% PhNH2 was pressured with 100 bar CO and 25 bar air and heated 1 h at 180° to give 72.6% PhNHCO2Et.  
ACCESSION NUMBER: 1982:598005 CAPLUS  
DOCUMENT NUMBER: 97:198005  
TITLE: Urethanes  
INVENTOR(S): Stammann, Guenter; Becker, Robert; Grolig, Johann; Waldmann, Helmut  
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.  
SOURCE: Eur. Pat. Appl., 37 pp.  
CODEN: EPXXDW  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 54218	A1	19820623	EP 1981-110031	19811201
EP 54218	B1	19841003		
R: BE, DE, FR, GB, IT				
DE 3046982	A1	19820715	DE 1980-3046982	19801212
JP 57126459	A2	19820806	JP 1981-197769	19811210
JP 02022744	B4	19900521		
US 4582923	A	19860415	US 1985-735249	19850517

PRIORITY APPLN. INFO.: DE 1980-3046982 A 19801212  
US 1981-328153 A1 19811207

L12 ANSWER 61 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
AB p-ClC6H4NO2 0.6, PhNH2 1.2, K2CO3 0.3, Cu oxide 0.075, and [Me(CH2)4]4N+.Cl- (I) 0.03 mol were stirred 10 h at 160° at 200-400 mm Hg, while removing the resulting H2O with PhNH2 and recycling PhNH2 to the reaction vessel to give 92.9% p-O2NC6H4NHPh, compared with 50.3% by reaction without I. RR13Z+.X- (R = PhCH2, Me; R1 = Et, octyl, Ph, or R = R1 = Bu, CMe3; Z = N, P; X = Cl, Br) were also used in place of I.  
ACCESSION NUMBER: 1982:438661 CAPLUS  
DOCUMENT NUMBER: 97:38661  
TITLE: Nitrodiphenylamine  
PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd., Japan  
SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.  
CODEN: JKKXAF  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

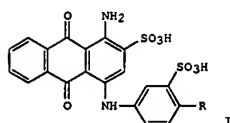
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 57040445	A2	19820306	JP 1980-115685	19800821

PRIORITY APPLN. INFO.: JP 1980-115685 A 19800821

L12 ANSWER 62 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Nitrophenylamines were prepared by reaction of halonitrobenzenes with anilines in the presence of acid scavengers, Cu catalysts, and polyethers under reduced pressures. Thus, 6 g com. polyether (average mol. weight 2000) was added to a mixture of 4-ClC<sub>6</sub>H<sub>4</sub>NO<sub>2</sub> 315, PhNH<sub>2</sub> 372, K<sub>2</sub>CO<sub>3</sub> 138, and CuO 6 g at 185-190° and 670-400 mm Hg with distillation of 4-5 mL/min aqueous PhNH<sub>2</sub> and recycling of distilled PhNH<sub>2</sub> to give, after 5 h, 93.8% (4-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>NH.  
 ACCESSION NUMBER: 1982:180948 CAPLUS  
 DOCUMENT NUMBER: 96:180948  
 TITLE: Nitrodiphenylamines  
 PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.  
 CODEN: JXXXXF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 57002243	A2	19820107	JP 1980-76844	19800606
JP 01010510	B4	19890222		
PRIORITY APPLN. INFO.:			JP 1980-76844	A 19800606

L12 ANSWER 63 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
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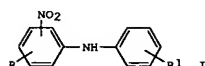


AB Anthraquinone acid dyes I (R = C<sub>10</sub>-15 alkyl) were prepared by treating 1-amino-4-bromoanthraquinone-2-sulfonic acid (II) [116-81-4] with 2,4-H<sub>2</sub>N(HO<sub>3</sub>S)C<sub>6</sub>H<sub>3</sub>R (III) in the presence of an acid-binding agent and Cu powder or a Cu salt. I gave wet- and light-fast blue shades on natural or synthetic polyamide fabrics. Thus, a solution containing 132 kg III [R = (CH<sub>2</sub>)<sub>11</sub>Me] [80822-87-3], prepared by sulfonation of 4-dodecylaniline [104-42-7] (H<sub>2</sub>SO<sub>4</sub>/oleum, <30°), together with Na<sub>2</sub>CO<sub>3</sub> 47, NaHCO<sub>3</sub> 56, and II 141 kg in 364 L water was heated to 40° under N, mixed with 4 kg CuCl, heated to 73° during 2 h, and maintained 4 h at 73-75° to give I [R = (CH<sub>2</sub>)<sub>11</sub>Me] [80822-88-4] which dyed polyamide fibers from a neutral-to-weakly acid dyebath.

ACCESSION NUMBER: 1982:105801 CAPLUS  
 DOCUMENT NUMBER: 96:105801  
 TITLE: Anthraquinone dyes  
 INVENTOR(S): Allen, Ernest Roy  
 PATENT ASSIGNEE(S): Imperial Chemical Industries Ltd., UK  
 SOURCE: Brit. UK Pat. Appl., 3 pp.  
 CODEN: BAXXDU  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 2061310	A	19810513	GB 1980-32461	19801008
PRIORITY APPLN. INFO.:			GB 1979-36850	A 19791024

L12 ANSWER 64 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
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AB Nitrodiphenylamines I (R, R<sub>1</sub> = H, alkyl) were prepared by reaction of RClC<sub>6</sub>H<sub>3</sub>NO<sub>2</sub> with R<sub>1</sub>C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> over Cu catalyst in the presence of K<sub>2</sub>CO<sub>3</sub> and m-caprolactam (II). Thus, a mixture of aniline 186, 4-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>Cl 157, CuO 2, xylene 86, K<sub>2</sub>CO<sub>3</sub> 69, and II 14 g was heated 10 h at 180-90° with removal of H<sub>2</sub>O to give 203.2 g I (R = R<sub>1</sub> = H, 4-NO<sub>2</sub>), vs. 135.5 g without II. Similarly prepared were I (R = R<sub>1</sub> = H, 2-NO<sub>2</sub>; R = H, R<sub>1</sub> = 4-Me, 4-NO<sub>2</sub>).  
 ACCESSION NUMBER: 1981:424516 CAPLUS  
 DOCUMENT NUMBER: 95:24516  
 TITLE: Nitrodiphenylamines  
 PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 3 pp.  
 CODEN: JXXXXF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 56022751	A2	19810303	JP 1979-99198	19790802
PRIORITY APPLN. INFO.:			JP 1979-99198	A 19790802

L12 ANSWER 65 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB The title reaction catalyzed by Cu salts on acidic carriers was used to prepare N-alkylated anilines and phenylenediamines. The catalyst suppressed hydrogenation of the ketones to secondary alcs. Thus, an autoclave was charged with 460 mL PhNH<sub>2</sub>, 740 mL Me<sub>2</sub>CO, and

30 g catalyst containing 18% Cu on a silica gel, pressurized with 70 atm H<sub>2</sub>, heated to 120°, and kept 280 min at 150° and 70-100 atmospheric. Conversion to PhNHCHMe<sub>2</sub> was 0.97.

ACCESSION NUMBER: 1980:110651 CAPLUS  
 DOCUMENT NUMBER: 92:110651  
 TITLE: Reduction alkylation of aromatic amines with aliphatic ketones  
 INVENTOR(S): Volf, Jiri; Pasek, Josef; Masek, Jan; Rezabek, Antonin  
 PATENT ASSIGNEE(S): Czech. Czech., 4 pp.  
 SOURCE: CODEN: CZXXA9  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Czech  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 179110	B	19790615	CS 1975-1557	19750310
PRIORITY APPLN. INFO.:			CS 1975-1557	19750310

L12 ANSWER 66 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
AB Aromatic amines were prepared by hydrogenating aromatic nitro or nitroso compds.  
over Cu-based catalysts, e.g., Cu chromite, with or without a promoter. Thus, hydrogenation of (4-O2NC6H4)2O over Cu chromite (Adkins type) gave 98% (4-H2NC6H4)2O of 99.8% purity.  
ACCESSION NUMBER: 1980:76081 CAPLUS  
DOCUMENT NUMBER: 92:76081  
TITLE: Aromatic amines  
INVENTOR(S): Culik, Vasile Cornel; Vlad, Virginia; Tolan, Mircea  
PATENT ASSIGNEE(S): Institutul de Cercetari si Proiectari Tehnologice pentru Rafinarii si Instalatii Petrochimice, Rom.  
SOURCE: Rom., 2 pp.  
CODEN: RUXXA3  
DOCUMENT TYPE: Patent  
LANGUAGE: Romanian  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:  

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
RO 64591	B	19780320	RO 1975-83083	19750805

  
PRIORITY APPLN. INFO.:  
RO 1975-83083 19750805

L12 ANSWER 67 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
AB p-O2NC6H4NHPh (I) was prepared by condensing PhNH2 with p-O2NC6H4Cl (II) in the presence of HCl acceptor (e.g., CuO-DMF or Cu) and 52% added Zn dust (based on II), followed by azeotropic drying. Thus, PhNH2 1.80, II 1.60, K2CO3 0.72, CuO 0.038, Zn dust 0.015 kg, xylene 200 and DMF 400 mL were heated to 184-6° and stirred at 8-9 h with azeotropic removal H2O, the mixture filtered and distilled in vacuo, leaving a residue containing 86.0% I (79.0-81.8 weight% yield). Steam distillation of the residue removed the residual II, leaving a mixture of I 92.0, (p-O2NC6H4)2NH 6.5, o-O2NC6H4NHPh 1.0 and aniline dye 0.5%.  
ACCESSION NUMBER: 1979:592970 CAPLUS  
DOCUMENT NUMBER: 91:192970  
TITLE: p-Nitrodiphenylamine  
INVENTOR(S): Szmajdja, Jerzy; Wilczek, Jerzy; Mikiciuk, Lucjan; Missala, Irena; Parulska-Szmajdja, Marianna; Sikorska, Alina  
PATENT ASSIGNEE(S): Instytut Przemyslu Organicznego, Pol.  
SOURCE: Pol., 3 pp.  
CODEN: POXXA7  
DOCUMENT TYPE: Patent  
LANGUAGE: Polish  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:  

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PL 101496	P	19781230	PL 1976-190443	19760614

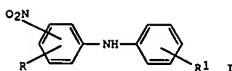
  
PRIORITY APPLN. INFO.:  
PL 1976-190443 A 19760614

L12 ANSWER 68 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
AB The title catalysts is a reaction product of N-methylpyrrolidone with a Cu compound, such as CuO, CuCN, 2CuCO3.Cu(OH)2. Thus PhNH2 93, 4-O2NC6H4Cl 157, CuO 2, and N-methylpyrrolidone 12 g was heated for 20 min at 160°, 93 g PhNH2 and 100 g K2CO3 added, and heated at 185° for 10 h to give 209 g 4-O2NC6H4NHPh. 4-O2NC6H4NHPh and 2-O2NC6H4NHPh were similarly prepared  
ACCESSION NUMBER: 1978:190365 CAPLUS  
DOCUMENT NUMBER: 88:190365  
TITLE: Catalyst and method for the preparation of nitrodiphenylamines  
INVENTOR(S): George, Joachim; Repplinger, Joachim  
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.  
SOURCE: Neth. Appl., 9 pp.  
CODEN: NAXXAN  
DOCUMENT TYPE: Patent  
LANGUAGE: Dutch  
FAMILY ACC. NUM. COUNT: 2  
PATENT INFORMATION:  

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
NL 7708331	A	19780131	NL 1977-8331	19770727
DE 2633811	A1	19780202	DE 1976-2633811	19760728
DE 2633811	C2	19831110		

  
PRIORITY APPLN. INFO.:  
DE 1976-2633811 A 19760728

L12 ANSWER 69 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
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AB Three title compds. I (R, R1 = H, alkyl), useful as intermediates for dyes or rubber stabilizers (no data), were prepared by treating R(O2N)C6H3Cl with R1C6H4NH2 in the presence of a Cu salt and 1-methylpyrrolidone. Thus, a mixture of PhNH2, 4-O2NC6H4Cl, CuO, xylene, and 1-methylpyrrolidone was refluxed 20 min at 160°, K2CO3 and PhNH2 added, and the mixture kept at 185° 10 h to give I (R = R1 = H, 4-NO2).  
ACCESSION NUMBER: 1978:152217 CAPLUS  
DOCUMENT NUMBER: 88:152217  
TITLE: Catalyst and its use in manufacturing nitrodiphenylamines  
INVENTOR(S): George, Joachim; Repplinger, Joachim  
PATENT ASSIGNEE(S): Bayer A.-G., Fed. Rep. Ger.  
SOURCE: Ger. Offen., 13 pp.  
CODEN: GWXXBX  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 2  
PATENT INFORMATION:  

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2633811	A1	19780202	DE 1976-2633811	19760728
DE 2633811	C2	19831110		
US 4122118	A	19781024	US 1977-818205	19770722
GB 1538039	A	19790110	GB 1977-30868	19770722
AU 7727332	A1	19790201	AU 1977-27332	19770726
AU 508633	B2	19800327		
CA 1088570	A1	19801028	CA 1977-283555	19770726
BE 857199	A1	19780127	BE 1977-179668	19770727
NL 7708331	A	19780131	NL 1977-8331	19770727
JP 53015366	A2	19780213	JP 1977-89386	19770727
JP 61037260	B4	19860822		
BR 7704933	A	19780328	BR 1977-4933	19770727
ES 461071	A1	19780601	ES 1977-461071	19770727
ZA 7704550	A	19780628	ZA 1977-4550	19770727
FR 2359825	A1	19780224	FR 1977-23305	19770728
FR 2359825	B1	19830121		

  
PRIORITY APPLN. INFO.:  
DE 1976-2633811 A 19760728

L12 ANSWER 70 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Cotton textiles were dyed fast shades by impregnating the textile with azo, triphenylmethane, or anthraquinone dyes containing a CO<sub>2</sub>H group attached directly to an aromatic ring, cyanamide [420-04-2] or dicyandiamide [461-58-5], and usually H<sub>3</sub>PO<sub>4</sub> and baking which leads to formation of an ester link between the dye and the cotton. Cotton textiles were also dyed by fixing a CO<sub>2</sub>H group-containing aniline derivative to the textile by the above methods, diazotizing, and coupling.

ACCESSION NUMBER: 1977:91665 CAPLUS  
 DOCUMENT NUMBER: 86:91665  
 TITLE: Reactively colored polymeric substrates, especially textile substrates  
 INVENTOR(S): McConnell, Bobby L.; Graham, Louis Atkins; Thornton, Raymond  
 PATENT ASSIGNEE(S): Burlington Industries, Inc., USA  
 SOURCE: Ger. Offen., 57 pp.  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 2  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2626836	A1	19761230	DE 1976-2626836	19760615
AU 503798	B2	19790920	AU 1976-15016	19760617
BE 843133	A1	19761220	BE 1976-168080	19760618
JP 52001188	A2	19770106	JP 1976-71256	19760618
FR 2316374	A1	19770128	FR 1976-18721	19760618
FR 2316374	B1	19800425		
BR 7603954	A	19770322	BR 1976-3954	19760618
CA 1078555	A1	19800603	CA 1976-255198	19760618
CH 630769	A3	19820715	CH 1976-7829	19760619
PRIORITY APPLN. INFO.:			US 1975-588840	A 19750620

L12 ANSWER 71 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB 4-PhNHC6H4NHCHRR1 (I; R = Me; R1 = C4-6 alkyl) were prepared by carboxylic acid-catalyzed condensation of 4-aminodiphenylamine (II) with RCOR1 followed by hydrogenation of the resulting Schiff bases. I were useful as stabilizers for synthetic rubber. Thus, a mixture of II 184, Me n-hexyl ketone 384, and stearic acid (III) 0.06 g was refluxed 4 hr with removal of H<sub>2</sub>O to give 291 g of Schiff base, which was autoclaved 5 hr at 150° with 5.9 g Cu chromite catalyst and 25-50 kg/cm<sup>2</sup> H to give 291 g I (R = Me, R1 = hexyl). I (R, R1 = Me, CH<sub>2</sub>CH<sub>2</sub>CHMe<sub>2</sub>; Me, Bu) were also prepared AcOH, salicylic acid, and BzOH were also used in place of III.

ACCESSION NUMBER: 1976:446174 CAPLUS  
 DOCUMENT NUMBER: 85:46174  
 TITLE: N-Phenyl-N'-alkyl-p-phenylenediamines  
 INVENTOR(S): Harada, Hiroshi; Ueno, Tsuneaki; Tanaka, Hiroaki; Nagai, Takayoshi  
 PATENT ASSIGNEE(S): Seiko Chemical Co., Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 51019732	A2	19760217	JP 1974-90093	19740806
PRIORITY APPLN. INFO.:			JP 1974-90093	A 19740806

L12 ANSWER 72 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB The polymerization of carboxylic acid anhydrides with diamines or diisocyanates, and carbonates gave hardenable polyamide-imide precursors useful for manufacture of heat and chemical resistant coatings with elec. insulating properties. Thus, a mixture of 4,4'-methylenedianiline 267.3, cresol (I) 700, trimellitic acid anhydride 288, and xylene 200 g was stirred at 170-200° separating H<sub>2</sub>O by azeotropic distillation, treated with 321 g diphenyl carbonate, heated for 5 hr at 240-60° separating PhOH by distillation, and precipitated in MeOH to give polyamide imide (II) [40009-12-9] with 0.13 intrinsic viscosity, 2.5 + 10-4 mole/g phenylurethane and 2.4 + 10-4 mole/g carboxyl groups. II solution in I was mixed with 78.3 g tris(2-hydroxyethyl) isocyanurate [839-90-7] and 5.10 g tetrabutyl titanate [5593-70-4], stirred for 20 min at 200°, cooled, and treated with 315 g C6H6 to give wire sheeting lacquer (30.8% solid and 35 P solution viscosity at 30°) which was coated on Cu wire, dried, and heated at 400-50° to give II-insulated wire with 42 rubbing resistant, 380° section temperature, and 16.5 kV/0.1 mm voltage breakdown.

ACCESSION NUMBER: 1975:98804 CAPLUS  
 DOCUMENT NUMBER: 82:98804  
 TITLE: Poly(amide imide) precursor-containing compositions  
 PATENT ASSIGNEE(S): Teijin Ltd.  
 SOURCE: Ger. Offen., 121 pp. Division of Ger. Offen. 2,239,611 (CA 79: 5876f).  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 7  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2264662	A1	19740801	DE 1972-2264662	19720811
DE 2264662	B2	19800925		
DE 2264662	C3	19810527		
JP 48026897	A2	19730409	JP 1971-61288	19710812
JP 54030034	B4	19790927		
JP 48039593	A2	19730611	JP 1971-73205	19710920
JP 51019479	B4	19760617		
JP 48039597	A2	19730611	JP 1971-73206	19710920
JP 51024000	B4	19760721		
JP 48042097	A2	19730619	JP 1971-75604	19710928
JP 51019478	B4	19760617		
JP 48043094	A2	19730622	JP 1971-76191	19710929
JP 51019480	B4	19760617		
JP 48079836	A2	19731026	JP 1972-10311	19720128
JP 51015859	B4	19760520		
PRIORITY APPLN. INFO.:			JP 1971-61288	A 19710812
			JP 1971-73205	A 19710920
			JP 1971-73206	A 19710920
			JP 1971-75604	A 19710928
			JP 1971-76191	A 19710929

L12 ANSWER 72 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)  
 JP 1972-10311 A 19720128



L12 ANSWER 73 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 GI For diagram(s), see printed CA Issue.  
 AB The CuSO<sub>4</sub>-catalyzed Ullmann condensation of Na 1-amino-4-bromo-9,10-dihydro-9,10-dioxoanthracene-2-sulfonate (I) with PhNH<sub>2</sub> in alkaline aqueous solution was examined. ESR studies showed that OH- and PhNH<sub>2</sub> were coordinated to Cu<sup>2+</sup>, Sn<sup>2+</sup>, Ti<sup>3+</sup>, and Fe<sup>2+</sup> accelerated the reaction. Anilines accelerated the condensation of I with p-MeC<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>NH<sub>2</sub>. The role of the Cu catalyst was discussed and a mechanism proposed.

ACCESSION NUMBER: 1974:504226 CAPLUS  
 DOCUMENT NUMBER: 81:104226  
 TITLE: Mechanism of the Ullmann condensation reaction. III. Role of the copper catalyst  
 AUTHOR(S): Tran Dinh Tuong; Hida, Mitsuhiro  
 CORPORATE SOURCE: Inst. Ind. Sci., Univ. Tokyo, Tokyo, Japan  
 SOURCE: Journal of the Chemical Society, Perkin Transactions 2: Physical Organic Chemistry (1972-1999) (1974), (6), 676-82  
 CODEN: JCPKBH; ISSN: 0300-9580  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English

L12 ANSWER 74 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 GI For diagram(s), see printed CA Issue.  
 AB The title compds. (I; R = H, Na; R1 = arylamino, cyclohexylamino) were prepared by reaction of the bromo compound (I; R = H, Na; R1 = Br) with R1NH<sub>2</sub> in the presence of Ti<sup>2+</sup> Sn<sup>2+</sup>, NaHSO<sub>3</sub> or glucose, an acid-neutralizing agent, and a Cu catalyst. Thus, I (R = Na, R1 = Br) 7.5, PhNH<sub>2</sub> 7, NaHCO<sub>3</sub> 3 parts were added to 100 parts H<sub>2</sub>O, the mixture heated under N to 70° and then treated with an aqueous solution of 0.1 part CuSO<sub>4</sub> and 0.1 part SnCl<sub>2</sub> and heated 30 min at 70° to give 94% I (R = Na, R1 = PhNH). Similarly prepared were 10 analogous I.

ACCESSION NUMBER: 1974:59797 CAPLUS  
 DOCUMENT NUMBER: 80:59797  
 TITLE: Anthraquinone compounds  
 INVENTOR(S): Hida, Mitsuhiro; Tabei, Itaru  
 PATENT ASSIGNEE(S): Nippon Kayaku Co., Ltd.  
 SOURCE: Jpn. Tokkyo Koho, 7 pp.  
 CODEN: JAXXAD  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 48014410	B4	19730507	JP 1969-93359	19691122
PRIORITY APPLN. INFO.:			JP 1969-93359	19691122

L12 ANSWER 75 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 GI For diagram(s), see printed CA Issue.  
 AB Nitrochlorobenzene and arylamines are condensed in solvents containing alkali carbonates, Cu catalysts, and HCONET<sub>2</sub>. Thus, p-nitrochlorobenzene, aniline, K<sub>2</sub>CO<sub>3</sub>, HCONET<sub>2</sub>, and CuI<sub>2</sub> and reagent class xylene are reacted 3 hr at 183-7° under stirring to give 92% 4-nitrodiphenylamine (I), m. 133°.

ACCESSION NUMBER: 1971:448665 CAPLUS  
 DOCUMENT NUMBER: 75:48665  
 TITLE: Nitrodiarylamines  
 INVENTOR(S): Itahashi, Shiro; Akiyoshi, Kuniyasu; Kawamoto, Shigeru; Takeshima, Yasuo  
 PATENT ASSIGNEE(S): Nippon Kayaku Co., Ltd.  
 SOURCE: Jpn. Tokkyo Koho, 3 pp.  
 CODEN: JAXXAD  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 46009452	B4	19710310	JP	19670428

L12 ANSWER 76 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Kinetic studies of the Ullmann condensation of Na 1-amino-4-bromo-2-anthraquinonesulfonate (I) and PhNH<sub>2</sub> were undertaken, using Cu (II) as catalyst. The reaction was first order in monomeric I and PhNH<sub>2</sub> and depended hyperbolically on the concentration of the catalyst. ESR measurements showed that the copper catalyst exists largely as Cu(II).

ACCESSION NUMBER: 1970:465587 CAPLUS  
 DOCUMENT NUMBER: 73:65587  
 TITLE: Mechanism of the Ullmann condensation. I. Kinetic and thermodynamic studies  
 AUTHOR(S): Tran Dinh Tuong; Hida, Mitsuhiro  
 CORPORATE SOURCE: Fac. Eng., Univ. Tokyo, Tokyo, Japan  
 SOURCE: Bulletin of the Chemical Society of Japan (1970), 43(6), 1763-8  
 CODEN: BCSJAB; ISSN: 0009-2673  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English

L12 ANSWER 77 OF 86 CAPLUS COPYRIGHT 2005 ACS ON STN  
AB Catalysts containing Mn and Ag on pumice carrier are employed in the catalytic reduction of 4-nitrosodiphenylamine to 4-aminodiphenylamine. Mn(OAc)2.4H2O (20.8 g) is dissolved in H2O and 15 ml concentrated HNO3 added. This solution is mixed with an aqueous solution of 13.4 g AgNO3, a 32% NaOH solution added, and the precipitate washed free of alkali by decanting. Fine pumice (particle size up to 0.085 mm) (100 g) is added to the residue and the solids filtered, washed with MeOH, Me2CO and dried at 100°. The product is then reduced with CO at 200-20° until no more CO2 is evolved. This catalyst (5 g) is mixed with 5 g 4-nitrosodiphenylamine in 100 ml water, and heated with H in an autoclave to 140° at 100 atm, to give a 74 mole % 4-aminodiphenylamine. Another catalyst employs a Ce-Cu coppt.  
ACCESSION NUMBER: 1970:414437 CAPLUS  
DOCUMENT NUMBER: 73:14437  
TITLE: Catalytic reduction of nitrosobenzenes to anilines  
INVENTOR(S): Dodman, David; Pearson, Kenneth W.; Wooley, John M.  
PATENT ASSIGNEE(S): Imperial Chemical Industries Ltd.  
SOURCE: Ger. Offen., 13 pp.  
CODEN: GWXXBX  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 1941008	A	19700219	DE 1969-1941008	19690812
GB 1241066	A	19710728	GB 1968-38708	19680813
NL 6912034	A	19700217	NL 1969-12034	19690807
FR 2019340	A5	19700703	FR 1969-27911	19690813
PRIORITY APPLN. INFO.:			GB 1968-38708	A 19680813

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GI For diagram(s), see printed CA Issue.  
AB The title compds. (I) were prepared by condensing (at 70-160°) 1 mole of a 2,5-dihaloterephthalic acid with 2 moles aryl amine, or a mixture of 2 amines, in a polar solvent in the presence of a Cu-containing catalyst, and an acid-binding agent. Thus, 11.8 parts 2,5-dichloroterephthalic acid, 15 parts anhydrous K2CO3, 55 parts ethylene glycol, 12.5 parts H2O, 76 parts aniline, 0.2 parts anhydrous CuAc2, and 2.5 parts KI were refluxed 5.5 hrs. at 120-125°. Addition of H2O and dilute HCl gave a precipitate, which was filtered off and purified by solution in dilute aqueous NaOH. Addition of Ac-OH gave I (R = R' = Ph); addition of HCl to the mother liquor gave 2-chloro-5-phenylaminoterephthalic acid. Preparation of 51 similar derivs. was described.  
ACCESSION NUMBER: 1963:461938 CAPLUS  
DOCUMENT NUMBER: 59:61938  
ORIGINAL REFERENCE NO.: 59:11350g-h, 11351a  
TITLE: 2,5-Diarylamino-terephthalic acids  
PATENT ASSIGNEE(S): Sandoz Ltd.  
SOURCE: 7 pp.  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 924661		19630501	GB	
CH 372316			CH	
CH 408952			CH	
US 3201402		1965	US	
PRIORITY APPLN. INFO.:			CH	19580729

L12 ANSWER 78 OF 86 CAPLUS COPYRIGHT 2005 ACS ON STN  
GI For diagram(s), see printed CA Issue.  
AB I, blue dyes for silk, wool, and synthetic polyamides, are prepared from 1-amino-4-bromoanthraquinone-2-sulfonic acid (II) or -2,6-disulfonic acid, and a substituted aniline in aqueous or aqueous alc. solution in the presence of a Cu catalyst and Na2CO3 or NaHCO3. When Y = H, I can be halogenated in concentrated H2SO4 or dilute oleum at 0-5°; the anilino ring may be sulfonated in H2SO4 with 25% oleum. A solution of 1,3-diamino-2,4,6-trimethylbenzene-5-sulfonic acid 92, Na2CO3 24, and H2O 200 parts was added to a mixture of II (Na salt) 81, Cu powder 2, CuCl 2, Na2CO3 32, and H2O 200 parts and the mixture heated at 50-5° for 20 hrs., then at 60-5° until II disappeared. The mixture was poured into 1000 parts H2O, 350 parts 30% HCl was added and the mixture filtered, washed with dilute HCl and pasted neutral with Na2CO3 to give I (R = X = H, V = SO3Na, W = Y = Me) (III), bright blue on silk, wool, and polyamides. A solution of 5.4 parts III in 50 parts H2O treated with 2 parts Ac2O and 3 parts NaHCO3 at 60-70° gave the III analog with R = Ac. Similarly, the following I were prepared (X, R, W, V, and Y given): H, CO2ClOH21, Me, SO3Na, Me; H, H, H, H, Me; H, Ac, SO3H, H, Me; H, Ac, H, Me; H, H, Me, NH2, Me; SO3Na, Ac, Me, H, H; SO3Na, Ac, Me, H, Br; SO3Na, H, Me, NH2, Me; SO3Na, H, Et, H, Et; SO3Na, COCH2CH2, Et, H, Et.  
ACCESSION NUMBER: 1966:68476 CAPLUS  
DOCUMENT NUMBER: 64:68476  
ORIGINAL REFERENCE NO.: 64:12851h, 12852a-c  
TITLE: Anthraquinone dyes  
INVENTOR(S): Peter, Albin; Baserga, Emilio; Guenthard, Jacques  
PATENT ASSIGNEE(S): Sandoz Ltd.  
SOURCE: 6 pp.  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CH 395397		19651231	CH	19580909

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AB (Throughout this abstract Pc = phthalocyanine). Cu and Ni Pc groups containing N-[4-((diaminotriazinyl)amino)phenyl] sulfonamide give washfast blue dyeings on cotton when applied (with or without prior methylation) with a resin-former and an acid catalyst and cured at 140°. Cu Pc-4-N-(3-amino-4-sulfonyl)sulfonamide sulfonic acid (I) 44.6 and cyanuric chloride 12.4 parts were allowed to react according to Brit. 805,562 (CA 53, 11851g) to give Cu Pc-4-N-[3-((4,6-dichloro-1,3,5-triazin-2-yl)amino)-4-sulfonyl)sulfonamide sulfonic acid (II), an aqueous solution of the Na salt of which was heated with concentrated aqueous NH3, 88 parts at 90° for 30 min., the mixture cooled to 20°, acidified with HCl, the precipitate washed with 2N HCl, suspended in H2O 1000 parts, brought to pH 7 with 2N NaOH and evaporated to dryness, giving a deep blue powder, (III), bright blue on cotton when applied with a resin-former and an acid catalyst and cured for 4 min. at 140°. Ni Pc-4-N-(3-amino-4-sulfonyl)sulfonamide sulfonic acid, and the 4-N-[4-((β-hydroxyethyl)amino)-3-sulfonyl], (4-methylamino-3-sulfonyl), and (3-amino-6-methyl-4-sulfonyl) analogs of I were also used to prepare similar dyes. II and CH2:CHCH2NH2, HN(CH2CH2OH)2, HO(CH2)4NH2 (x = 3,4,5), or MeNHCH2CH2OH gave similar dyes. A solution of III 15.2 in 36.7% aqueous HCHO 50 parts, adjusted to pH 8 with 2N NaOH, heated for 30 min. at 70°, cooled to 20°, and precipitated by addition of Me2CO 300 parts gave a dye containing 5 CH2OH groups per triazine residue, bright blue on cotton; reaction of III with HCHO at 20° gave a monomethylol derivative  
ACCESSION NUMBER: 1963:15218 CAPLUS  
DOCUMENT NUMBER: 58:15218  
ORIGINAL REFERENCE NO.: 58:2529f-h, 2530a  
TITLE: Phthalocyanine aminotriazine dyes  
INVENTOR(S): Clark, Peter F.; Howard, Harold T.  
PATENT ASSIGNEE(S): Imperial Chemical Industries Ltd.  
SOURCE: 9 pp.  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 904227		19620822	GB	19590527

L12 ANSWER 81 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
AB Copper chromite has been investigated as a catalyst  
for the preparation of alkylaryl secondary amines by the reductive  
alkylation  
of a primary aromatic amine with an aliphatic ketone in the presence of  
hydrogen. Copper chromite avoids the nuclear hydrogenation and  
hydrogenolysis of carbon-nitrogen bonds obtained with Ni and with noble  
metal catalysts; a large amount of ketone is reduced to the  
corresponding alc. The desired selectivity, i.e., reductive alkylation  
rather than ketone reduction, is favored by low pressure.  
ACCESSION NUMBER: 1962:462418 CAPLUS  
DOCUMENT NUMBER: 57:62418  
ORIGINAL REFERENCE NO.: 57:12353c-d  
TITLE: Copper chromite catalysts for  
reductive alkylation  
AUTHOR(S): Dovell, Fred S.; Greenfield, Harold  
CORPORATE SOURCE: U. S. Rubber Co., Naugatuck, CT  
SOURCE: Industrial & Engineering Chemistry Product Research  
and Development (1962), 1, 179-81  
CODEN: IEPRA6; ISSN: 0196-4321  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable  
OTHER SOURCE(S): CASREACT 57:62418

L12 ANSWER 82 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
AB cf. CA 54, 2224h. Preparation of a series of  
1-picryl-2,2-diphenylhydrazyl  
free radicals substituted in the p-positions of the 2 benzene rings by  
OMe, Me, F, Cl, Br, CO<sub>2</sub>Me, NO<sub>2</sub>, or Ph groups was described. Some  
limitations in the conventional preparative sequence used for  
1-picryl-2,2-diphenylhydrazyl (I) was revealed in this series of compds.  
Two new reagents, NOCl for N-nitrosation of diarylamines, and  
N-picrylpyridinium chloride for picrylation of 1,1-diarylhydrazines,  
offered advantages at these stages of the preparative sequence. Some  
alternative methods which involved substitution reactions with various  
compds. of that series were also developed. N-Acetylarylamine (0.5 mole),  
0.5 mole aryl iodide, 50 ml. xylene, 0.5 formula weight K<sub>2</sub>CO<sub>3</sub>, 10 g.  
Fernlock  
Cu powder, and a few crystals of iodine were heated 48 hrs. at  
200° or longer, the mixture cooled, 1500 ml. 10% alc.NH<sub>4</sub>OH added, the  
mixture refluxed 8-15 hrs., the solvents removed, and the solid residue  
extracted with ligroine in a Soxhlet apparatus, the extract  
concentrated, cooled, and the  
crystalline solids collected. If the product was tarry it was subjected  
to  
further treatment in the Soxhlet apparatus The following secondary  
aromatic  
amines were thus obtained (p-group., extraction solvent, recrystn.  
solvent,  
m.p., and % yield given): OMe, hexane, C<sub>6</sub>H<sub>6</sub>-hexane, 100-2°, 46; Me,  
hexane, hexane, 82-3°, 60; F, none, none, b.d. 123-5°, 71;  
Cl, hexane, hexane, 77-8°, 51; CO<sub>2</sub>Me, CHCl<sub>3</sub>, C<sub>6</sub>H<sub>6</sub>, 178-80°,  
42; Ph, C<sub>6</sub>H<sub>6</sub>, C<sub>6</sub>H<sub>6</sub>, 209-10°, 32. Bromination at 40° of a  
CHCl<sub>3</sub> solution of N-benzoyldiphenylamine (II) gave 69% bis(p-  
bromophenyl)amine, (II), m. 105.5-7.0° (hexane). II (0.1 formula  
weight) in AcOH treated dropwise at 90-5° with fuming HNO<sub>3</sub> gave 38%  
bis(p-nitrophenyl)amine, m. 217-18°. Nitrosation was carried out  
by adding 1.2 formula wts. NaNO<sub>2</sub> in 250 ml. H<sub>2</sub>O to 1 formula weight of  
the  
amine in 500 ml. alc. and 100 ml. HCl at 0°, stirring the mixture an  
addnl. 0.5 hr., keeping overnight at room temperature, and evaporating  
Reaction with  
diphenylamine gave 95% nitroso compound, m. 65-6°. It gave  
N-nitrosodi-p-anisylamine in 60% yield, m. 78-80°. It was also  
used to nitrosate the amines substituted by other groups. The following  
substituted secondary N-nitrosoamines were thus obtained (p-group,  
nitrosation reagent, recrystn. solvent, m.p., and % yield given): F,  
HNO<sub>2</sub>,  
hexane, 45-6.5°, 90; Cl, HNO<sub>2</sub>, hexane, 81-2.5°, 81; Br,  
HNO<sub>2</sub>, hexane, 104-6°, 80; CO<sub>2</sub>Me, HOCl, MeOH, 105-7°, 80;  
NO<sub>2</sub>, HOCl, MeNO<sub>2</sub>, 145-8°, major part decomposed during attempted  
recrystn. from MeNO<sub>2</sub>; Ph, NOCl, alc., 155-80°, a mixture Those  
nitrosoamines substituted by electron withdrawing groups were appreciably  
less stable than N-nitrosodiphenylamine. N-Nitrosobis(p-  
carbomethoxyphenyl)amine was converted to the free amine by sublimation  
at  
100° or heating the molten nitroso compound N-Nitrosobis(p-  
nitrophenyl)amine was also formed by the NOCl, and a small amount  
isolated  
by extraction with hexane. N-Nitrosoamines were reduced by LiAlH<sub>4</sub> in  
Et<sub>2</sub>O.  
The reduction gave 90% 1,1-di-p-anisylhydrazine, m. 110-11° (MeOH).  
The following substituted 1,1-diarylhydrazines were thus obtained  
(p-groups, recrystn. solvent, m.p., and % yield given): F, alc.,

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185-6°, -; Cl, hexane, 92.5-3.5°, 72; Br, alc.,  
117-19°, 89; CO<sub>2</sub>Me, MeOH, 169-72°, 17; Ph, alc.,  
226-7.5°, 34. II (0.022 formula wt.) in 200 ml. Et<sub>2</sub>O stirred 15  
min. with BuLi in pentaneheptane, the mixt. poured on 100 g. crushed dry  
ice, left overnight, extd. into 10% NaOH, the ext. acidified, the solid  
recovered, suspended in Et<sub>2</sub>O, treated with CH<sub>2</sub>N<sub>2</sub>, and the product  
recovered and recrystd. gave 1,1-bis(p-carbomethoxyphenyl)hydrazine.  
1,2-Dichloroethane (150 ml.) and 0.3 formula wt. AlCl<sub>3</sub> at 10°  
treated with 26 ml. AcCl, followed by 0.03 mole diphenylhydrazine-HCl,  
the  
mixt. stirred overnight at room temp., poured on ice, the solvent steam  
distd., and the crude residue recrystd. gave 60%  
1,1-bis(p-acetylphenyl)-2-  
acetylhydrazine, m. 193-4° (alc.). Addn. of an equimolar amt. of  
dry C<sub>5</sub>H<sub>5</sub>N to picryl chloride in CHCl<sub>3</sub> formed the reagent. The  
diarylhydrazine (0.05 formula wt.) in 100 ml. CHCl<sub>3</sub> added to a suspension  
of an equiv. amt. of the above reagent in 250 ml. CHCl<sub>3</sub>, the mixt.  
stirred  
at room temp. until the color became deep red, the suspended pyridinium  
chloride filtered off, the filtrate washed with H<sub>2</sub>O, dried, and evapd.  
The following substituted 1,1-diaryl-2-picrylhydrazines were thus  
obtained  
(p-group, reaction time in hrs., recrystn. solvent, m.p., and % yield  
given): OMe, 0.8, Ph, 92-5°, 39; Me, 4, 2:1 CHCl<sub>3</sub>-alc.,  
166-7°, 80; H, 20, 3:2 CHCl<sub>3</sub>-alc., 174-6°, 95; F, 6, 3:2  
CHCl<sub>3</sub>-alc., 196-8°, 92; Cl, 20, CCl<sub>4</sub>, 165-6°, 73; Br, 24,  
CCl<sub>4</sub>, 107.5-9.5°, 90; CO<sub>2</sub>Me, 46, CHCl<sub>3</sub>, 184-8°, 45; NO<sub>2</sub>,  
0.5, dioxane, 212-13.5°, 75; Ph, 24, CHCl<sub>3</sub>-CCl<sub>4</sub>, 121-3°, 38.  
Attempts to prep. 1,1-di-p-anisyl-2-picrylhydrazine (III) under the  
conditions described above gave large ams. of a green decompn. product.  
Better results were obtained by starting the picrylation in a cold soln.  
Dianisylhydrazine (0.067 formula wt.) in 160 ml. CHCl<sub>3</sub> treated at  
3° with 0.067 formula wt. N-picrylpyridinium chloride in 240 ml.  
CHCl<sub>3</sub>, the mixt. stirred 10 min. and an addnl. 40 min. at room temp., the  
soln. evapd., the residue taken up in C<sub>6</sub>H<sub>6</sub>, and the soln. chromatographed  
on Al<sub>2</sub>O<sub>3</sub> and crystd. gave red crystals of III, m. 92-5°. When the  
C<sub>6</sub>H<sub>6</sub> was completely removed the residue melted at 64-7°. Later  
fractions which were allowed to stand or heated above room temp. were  
green. These were believed to be the dianisylnitrogen radical. The more  
basic product was pptd. as the HCl salt, then condensed with benzil. The  
m.p. of 259-60° corresponded to that reported for the benzil  
condensation product of 1,2,3,5-tetraaminobenzene. Attempts to hydrolyze  
1,1-bis(p-nitrophenyl)-2-benzoylhydrazine in H<sub>2</sub>SO<sub>4</sub> of various concns.,  
HCl, or in H<sub>3</sub>PO<sub>4</sub> gave no reaction or a tarry product.  
1,1-Bis(p-nitrophenyl)-2-picrylhydrazine was not isolated. An attempt  
was  
made to prep. the picrylhydrazine directly. 1-Picryl-2-p-  
nitrophenylhydrazine was obtained in 92% yield, m. 214-15°, by  
reaction of N-picrylpyridinium chloride and p-nitrophenylhydrazine in  
CHCl<sub>3</sub>. This compd. in refluxing alc. treated with Cu(OAc)<sub>2</sub> gave  
2,4,4',6'-tetranitroazobenzene, m. 171-3°. The  
diarylpicrylhydrazines were oxidized by stirring in CCl<sub>4</sub> or CHCl<sub>3</sub> solns.  
with excess PbO<sub>2</sub> in the presence of anhyd. Na<sub>2</sub>SO<sub>4</sub> with the reaction time  
varying from 0.25 to 2 hrs., the inorg. compds. were removed, and the  
filtrate evapd. In most cases crystn. began at the end of the evapn.  
The  
following 1-picryl-2,2-diarylhydrazyl free radicals were obtained  
(p-group, hydrazine (mole), PbO<sub>2</sub> (mole), g. of Na<sub>2</sub>SO<sub>4</sub>, solvent (ml.),  
reaction time in hrs., soln. color, purification, and m.p. given): OMe,

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0.0022, 0.11, 15, CHCl<sub>3</sub> (50), 0.25, blue, none, 145-8°, CH<sub>3</sub>,  
0.0024, 0.11, 20, CHCl<sub>3</sub> (50), 0.33, blue-purple, lyophilized from C<sub>6</sub>H<sub>6</sub>,  
108-18°; H, 0.0051, 0.051, 0, CCl<sub>4</sub> (60), 1.0, purple, recrystd.  
from CCl<sub>4</sub>, 127-9°; Cl, 0.0065, 0.053, 7.2, CCl<sub>4</sub> (180), 1.0, purple,  
from CCl<sub>4</sub>, 154-6°; Br, 0.011, 0.22, 15, CCl<sub>4</sub> (300), 1.0, purple,  
from CCl<sub>4</sub>, 139-41°; CO<sub>2</sub>Me, 0.001, 0.08, 20, CHCl<sub>3</sub> (70), 1.0, dull  
purple, lyophilized from C<sub>6</sub>H<sub>6</sub>, 90-100°; NO<sub>2</sub>, 0.0043, 0.15, 17,  
CHCl<sub>3</sub> (80), 2.0, brown-purple, from EtOAc, 204-7°; Ph, 0.0042,  
0.05, 6, CHCl<sub>3</sub> (150), 1.0, blue-purple, lyophilized from CHCl<sub>3</sub>,  
140-50°. The substituted radicals seemed to be the more stable.  
Solns. of the radicals decompd. slowly at rates which increased with  
substitution by stronger electron-accepting groups. There was no  
evidence  
of dimerization in soln. No satisfactory method was devised for  
recrystn.  
of the above radicals. All of the reasonably stable radicals except that  
substituted by Ph groups gave good analyses.  
ACCESSION NUMBER: 1961:143816 CAPLUS  
DOCUMENT NUMBER: 55:143816  
ORIGINAL REFERENCE NO.: 55:27166e-1,27167a-1,27168a-b  
TITLE: Preparation of substituted 1-picryl-2,2-  
diphenylhydrazyl free radicals  
AUTHOR(S): Chen, Mabel M.; D'Adamo, Amedeo F., Jr.; Walter,  
Robert I.  
CORPORATE SOURCE: Haverford Coll., Haverford, PA  
SOURCE: Journal of Organic Chemistry (1961), 26, 2721-7  
CODEN: JOCEAH; ISSN: 0022-3263  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable  
OTHER SOURCE(S): CASREACT 55:143816

L12 ANSWER 83 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB In the preparation of the named compds. by reaction of an aromatic amino, nitro, or nitroamino compound with H and a lower-dialkyl ketone (e.g. MeCOEt), an aqueous distillation cut (I) from the final product comprising the H<sub>2</sub>O-ketone azeotrope is recycled to the feed. Preferably the starting compound is dissolved in the I, any H<sub>2</sub>O that separates is discarded, and the remaining solution is used as feed for the reaction. A suitable catalyst is Cu deposited on Cr<sub>2</sub>O<sub>3</sub> (or on ZnO, Al<sub>2</sub>O<sub>3</sub>, C, etc.), preferably pelleted at 15 tons/sq. in.

ACCESSION NUMBER: 1959:11660 CAPLUS  
 DOCUMENT NUMBER: 53:11660  
 ORIGINAL REFERENCE NO.: 53:2156h-i  
 TITLE: Alkylated aromatic amines  
 INVENTOR(S): Lamb, Sidney A.; Ward, Stanley  
 PATENT ASSIGNEE(S): Imperial Chemical Industries Ltd.  
 SOURCE: Addn. to Brit. 716,239 (C.A. 49, 14804c)  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 793716		19580423	GB	

L12 ANSWER 84 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB Aminoazo compds. were prepared and reductively alkylated to amines by catalytic hydrogenation in the presence of ketones. E.g., 22.4 g. aniline (I) in 150 ml. MeOH and 52 ml. concentrated HCl diazotized at 0-2° with 20 g. NaNO<sub>2</sub> in 25 ml. H<sub>2</sub>O, cooled to -10°, added to 33.8 g. Ph<sub>2</sub>NH (II) in 500 ml. MeOH and 52 ml. concentrated HCl. previously cooled to 8-10°, the mixture kept 72 hrs. at about -15° and the p-PhNHC<sub>6</sub>H<sub>4</sub>N<sub>2</sub>Ph (III) solid HCl salt filtered off, washed with H<sub>2</sub>O, and made basic with aqueous NH<sub>3</sub>, washed further with H<sub>2</sub>O, and dried yielded 44.3 g. III, m. 85-6°. III in Et<sub>2</sub>O dried over Na<sub>2</sub>CO<sub>3</sub>, filtered, and the Et<sub>2</sub>O removed by distillation left III substantially free of Cl. III (850 g.) in 3400 g. acetone passed over 1 l. of a Cu chromite catalyst maintained at 165° at the rate of 680 g./hr. and H at a space velocity of 550 l./hr., with the pressure maintained at 50 atmospheric, and the acetone, iso-PrOH, and H<sub>2</sub>O stripped off left 911 g. crude product which on vacuum distillation yielded 269 g. of PhNHC<sub>6</sub>H<sub>4</sub>Me<sub>2</sub> and 561 g. p-PhNHC<sub>6</sub>H<sub>4</sub>CHMe<sub>2</sub>. III (44 g.) in 500 ml. EtCOME reductively alkylated with 1 g. of a 30% by weight Pt on C catalyst 8 hrs. at 80° then 16 hrs. at 160° and a maximum of 100 atmospheric H yielded 22.1 g. PhNHC<sub>6</sub>H<sub>4</sub>Me<sub>2</sub> and 33.3 g. 4-(PhNHC<sub>6</sub>H<sub>4</sub>)NHC<sub>6</sub>H<sub>4</sub>Me<sub>2</sub>, m. 49-50°. Hydrogenation of 100 g. III in 500 ml. cyclohexanone 6 hrs. at 60° and 24 hrs. at 160° with a Pt-on-C catalyst yielded 86.5 g. 4-cyclohexylaminodiphenylamine, m. 110-14°. I (17 g.), 100 ml. MeOH, and 50 ml. concentrated HCl at 0° diazotized with 12.7 g. NaNO<sub>2</sub> in H<sub>2</sub>O, added to 30 g. (m-MeC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>NH, 80 ml. MeOH, and 50 ml. concentrated HCl with the temperature maintained at -10°, the solid filtered off after 45 min., washed, treated with aqueous NH<sub>3</sub>, extracted with Et<sub>2</sub>O, and the Et<sub>2</sub>O evaporated gave 24.8 g. 2-methyl-4-(3-methylphenylamino)azobenzene (IV), m. 99°, hydrogenated in EtCOME to 3,3'-dimethyl-4-sec-butylamin-diphenylamine, p-Toluidine in MeOH diazotized, added to II in MeOH and HCl, and kept 16 hrs. at -10° gave 4-(4-PhNHC<sub>6</sub>H<sub>4</sub>N<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>Me (V), m. 116-18° (from petr. ether), hydrogenated in EtCOME to p-MeC<sub>6</sub>H<sub>4</sub>NHC<sub>6</sub>H<sub>4</sub>Me<sub>2</sub> and 4-PhNHC<sub>6</sub>H<sub>4</sub>NHC<sub>6</sub>H<sub>4</sub>Me<sub>2</sub>, m. 46-7°. m-Toluidine in place of p-toluidine gave 3-(4-PhNHC<sub>6</sub>H<sub>4</sub>N<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>Me, m. 68-8.5°. p-Anisidine gave 4-(4-PhNHC<sub>6</sub>H<sub>4</sub>N<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>OMe, m. 112°.

ACCESSION NUMBER: 1957:81684 CAPLUS  
 DOCUMENT NUMBER: 51:81684  
 ORIGINAL REFERENCE NO.: 51:14799g-i,14800a-b  
 TITLE: Amines  
 INVENTOR(S): Dent, Walter T.  
 PATENT ASSIGNEE(S): Imperial Chemical Industries Ltd.  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
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PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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L12 ANSWER 85 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN  
 AB The 85-15 copolymers of methyl methacrylate-maleic anhydride react with alcs., amines, and metallic salts to produce clear products which are solvent-, heat-, and abrasion-resistant: Esterification of 85-15 copolymer and primary and secondary aromatic and aliphatic alcs. react to produce half-esters at 70° in the presence of 0.5% Na<sub>2</sub>CO<sub>3</sub> catalyst. The products are softer than the original. Resistance to solvents and heat is increased when ethylene glycol, glycerol, and diethylene glycol are used. Amination, solns. of aniline, o- and p-chloroaniline, p-bromoaniline, o- and p-aminophenol, 2-amino-4-nitrophenol, p-aminodimethylaniline, sulfanilic acid, p-aminobenzoic acid, p-aminobenzenesulfonamide, p-aminobenzophenone, p-aminobiphenyl, 2-amino-p-cymene, 1-aminoanthraquinone, α-naphthylamine, 2,4-diaminodiphenylamine, dibutylamine, and octadecylamine in aqueous or anhydrous MeOH produce materials which have increased shear hardness, equivalent mar resistance, but sometimes colored or crazed surfaces compared with the original copolymer. Metal salts, with cations of H, NH<sub>4</sub><sup>+</sup>, Ti<sup>+++</sup>, Ag, Cu<sup>++</sup>, K, Zn, Mg, Mn<sup>++</sup>, Ca, Cd, Ni<sup>++</sup>, Ba, Sn<sup>++</sup>, Al, Cr<sup>+++</sup>, Hg<sup>++</sup>, Fe<sup>+++</sup>, Co<sup>++</sup>, Cu (NH<sub>3</sub>)<sub>4</sub><sup>++</sup>, Sr<sup>++</sup>, UO<sub>2</sub><sup>++</sup>, show generally improved shear hardness, scratch resistance and greater percentage transmission than the original copolymers.

ACCESSION NUMBER: 1949:45509 CAPLUS  
 DOCUMENT NUMBER: 43:45509  
 ORIGINAL REFERENCE NO.: 43:8202a-e  
 TITLE: Surface reactions of copolymers. Methyl methacrylate and maleic anhydride  
 AUTHOR(S): Seymour, Raymond B.; Branum, Ira, Jr.; Hayward, F. W.  
 SOURCE: Journal of Industrial and Engineering Chemistry (Washington, D. C.) (1949), 41, 1482-4  
 CODEN: JIECAD; ISSN: 0095-9014  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Unavailable

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 AB The extent of polymerization at the gel point of monomers containing allyl and methacrylate groups can be increased (1) by diminishing the mol. weight or (2) by diminishing the tendency of one of the unsatd. groups to enter into the copolymer. Polymerization of allyl methacrylate (I) in presence of additives: In preliminary expts. the polymerization of I was examined in the presence of low concns. of inhibitors, retarders, or chain-transfer reagents, and the effects of these compds. on time of gelation and extent of reaction at the gel point were observed. Solns. of the peroxide and the additive in I, sealed in vacuum in glass tubes, were rotated in an oil bath at 75 ± 0.5° until gelation occurred, the time of gelation and n of the product were noted, and from the latter the extent of conversion was calculated. The concentration of the additive, except where otherwise indicated, was 0.006 mole/l.; the concentration of Bz2O2 was 0.0124 or 0.0248 mole/l. (the latter indicated by \*). Results (data in moles/l.): (1) Compds. which do not affect the time and extent of reaction at gelation: 2-mercaptobenzothiazole (II) \*, 0.01; thiobarbituric acid (III) \*, 0.002; thiourea (IV) \*, 0.003; dithiodiglycolic acid; CS2\*, 0.04; biacetyl, 0.018; benzaldehyde\*, 0.046; β-iodopropionic acid\*, 0.026; α,α'-dibromo-p-xylene\*, 0.075; p-nitrosodimethylaniline; 2,4-dinitrophenylhydrazine; dimethyl glyoxime; α-benzildioxime. (2) Compds. which are unsatisfactory, extending the time for gelation 10-fold or more without leading to appreciable increase in extent of reaction at gel point: tetraethylthiuram disulfide\*, 0.04; hydroquinone monoethyl ether; 2,5-di-tert-butylhydroquinone\*; hydroquinone; quinhydrone; quinone; Cu acetylacetonate; ethylenediamine; α-nitroso-β-naphthol (significant increase in extent of reaction at gel point with excessive inhibition). (3) Materials which extend the gel time moderately and do not increase the extent of polymerization at the gel point are: p-nitrosodimethylaniline; phenol; di-tert-butylquinone\*; alr; Cu methacrylate. (4) Compds. which increase gel time and extent of polymerization at the gel point are: α-naphthylamine; Akroflex C; 2,4-dinitrophenylhydrazine; 2,4-dinitrophenol; chloranil; β-thionaphthol (V). Because of an interest in additives which did not affect the color of the polymer and because V showed the most marked effect in increasing the extent of reaction at the gel point, the behavior of mercaptans and diisopropyldixanthogen (VI) on the gelation of I was examined further with these results at 75° (in the order given): additive, concentration of additive (mole/l.), Bz2O2 (mole/l.), gel point: time (min.) and polymer % (from nD20): no additive -, 0.012, 8, 6; V, 0.006, 0.012, 27, 10; V, 0.016, 0.012, 35, 15; V, 0.025, 0.012, 40, 21; V, 0.063, 0.012, 120, 23; thiophenol (VII) 0.017, 0.012, 45, 15; β-mercaptoethanol (VIII), 0.250, 0.012, > 325, > 23; lauryl mercaptan (IX), 0.058, 0.025, 30, 16; IX, 0.092, 0.025, 65, 20; thioglycolic acid (X), 0.043, 0.083, 20, 28; X, 0.034, 0.012, 48, 15; X, 0.079, 0.025, 55, 26; X, 0.015, 0.025, 70, 34; VI, 0.022, 0.062, 14, 19; VI, 0.040, 0.025, 21, 17; at 50° and diisopropyl peroxydicarbonate (0.033 mole/l.) X, 0.025, 21, 17; at 50° and diisopropyl peroxydicarbonate (0.033 mole/l.) X,

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 copolymers and on mol. wt.: If M1 and M\*1 = a methacrylate group and methacrylate-derived radical, resp., M2 and M\*2 = an allyl group and allyl-derived radical, R and R\* = the regulator mol. and the radical derived from it, and P a polymer, then the reactions occurring in the polymerization of I in the presence of a regulator can be indicated thus: M\*1 + M1 → M\*1 (1) k11 M\*1 + R → P + R\* (5) k1R M\*1 + M2 → M\*2 (2) k12 M\*2 + R → P + R\* (6) k2R M\*2 + M2 → M\*2 (3) k22 R\* + M1 → M\*1 (7) kR1 M\*2 + M1 → M\*1 (4) kR2 R\* + M2 → M\*2 (8) kR2 While a prediction of the effect of the regulator on the compn. of the copolymer is difficult, the following is suggested: (1) If the mol. wts. are fairly high, the methacrylate monomer may be consumed largely via equations 1 and 4, and the allyl monomer via equations 2 and 3. If either monomer is reacting slowly, equations 7 and 8 may be important. The regulator cannot change the rate constn. or monomer concns. of reactions 1 to 4 and affects their rates and relative rates only by changing the concns. of M\*1 and M\*2 via reactions 5 to 8. Sets of conditions whereby the regulator changes the relative concns. of M\*1 and M\*2 are considered: If k2R > k1R and kR1 kR2, the regulator increases the steady state ratio of M\*1/M\*2, and if M\*2 has higher probability of reacting with M2 than has M\*1, decreased incorporation of M2 into polymer results. Such effects are small. The effect of the regulators in lowering the mol. wt. seems to be largely due to chain transfer reactions. Postponement of gelation: The I system was discussed in detail by Cohen, et al. (C.A. 42, 5256g). The 4-fold diminution in mol. wt. in the copolymerization of XIII and XI in the presence of 0.07 mole/l. X can be compared qualitatively with a similar increase in extent of reaction at the gel point in the polymerization of I in the presence of 0.07 mole/l. X. The apparent failure of X to increase the extent of reaction at the gel point in dimethacrylate polymerization may indicate that in such polymers with very high probability of cross linkage a much greater diminution in chain length is needed to affect the gel point. The apparent lack of effectiveness in postponing diallyl monomer gelation may result from the fact that in polymers with high initiator requirements chain lengths are already low and further diminution in chain length by small amts. of mercaptan is relatively unimportant. Preliminary expts. indicate that the presence of 0.04 mole/l. X caused only a slight decrease in mol. wt. in the polymerization of XIII.

ACCESSION NUMBER: 1949:45504 CAPLUS  
 DOCUMENT NUMBER: 43:45504  
 ORIGINAL REFERENCE NO.: 43:8199c-i, 8200a-i, 8201a-e  
 TITLE: Action of regulators in polymerization of monomers containing allyl and methacrylate groups  
 AUTHOR(S): Cohen, Saul G.; Sparrow, Donald B.  
 SOURCE: Journal of Polymer Science (1948), 3, 693-703  
 CODEN: JPSCAU; ISSN: 0022-3832  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Unavailable

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 0.066, -, 55, 20. These results show that the extent of reaction at the gel point appears to be increased with increasing concn. of the peroxide and the mercaptan. By proper choice of conditions the extent of reaction at the gel point can be increased from 6 to about 28% polymerization. The polymerization of other polyfunctional monomers, Du Pont BCM, a glycol dimethacrylate, and diallyl phthalate in the presence of 0.04-0.08 mole/l. X showed little change in the extent of polymerization at the gel point. Polymerization of methyl methacrylate (XI), in the presence of additives: The comparative effects of several types of S-contg. compds. were studied by heating a soln. of 3.71 mole/l. XI and 0.025 mole/l. Bz2O2 in AcOEt at 75.0 ± 0.1° for 1 hr. in evacuated sealed tubes with sufficient additive to form a 0.03 mole/l. soln. The extent of polymerization was estd. by titration for unsatn., the polymer was pptd. by CH3OH, and the intrinsic η was detd. in C6H6 soln. at 30°. The following are the % reaction and nsp/c found at 75° for the additives given: none, 36, 0.30; III, 33, 0.32; II, 33, 0.34; X, 31, 0.14; VI, 31, 0.18; IV, 7, 0.19; urea, 36, 0.30. Only the mercaptan and the dixanthogen would be useful regulators under these exptl. conditions. Characterization of sol. polyallyl methacrylate (XII): The bromination of sol. XII was examd. to est. its content of unsatd. allyl and methacrylate groups. A sample of I contg. Bz2O2 and X, 0.083 and 0.043 mole/l., resp., was heated at 75° for 15 min.; nD20 1.4510, 18% polymer. Another sample contg. 0.0124 mole/l. Bz2O2 and no X heated at 75° for 5 min. gave a polymer content of 4%. In both cases the catalyst was destroyed, the polymer was pptd. by CH3OH, repptd. from acetone, dried, and a sample was analyzed for unsatn. by bromide-bromate titration in 1:1 CCl4-AcOH under conditions under which allyl acetate consumes 97-98% of the theoretical amt. of Br while XI consumes only 45% of the theoretical amt. of Br. The 2 samples of sol. XII prepd. in the presence and absence of X consumed 97 and 96%, resp., of the theoretical amt. of Br, were practically identical, and contained very little methacrylate unsatn. Two samples of sol. XII prepd. in the absence and presence of mercaptan were isolated after 4 and 14% polymerization and showed an intrinsic η of 2.0 and 0.20, resp. The decrease in the mol. wt. was greater than could be accounted for by the difference in peroxide concn. and was due largely to the mercaptan; while the latter did not appreciably alter the comparative rates of entry into the polymer of allyl and methacrylate groups it diminished the mol. wt. markedly. Copolymerization of allyl chloroacetate (XIII) and XI: The Bz2O2-initiated copolymerization of XIII and XI (concns. 5.06 and 3.73 moles/l., resp.) in the presence and absence of X was studied by carrying out the reactions in vacuo in rotating sealed tubes at 75° ± 0.1°, titrating for unsatn. initially and after stated periods of heating, isolating polymer samples by pptn. with CH3OH, and examm. for mol. wt. and Cl content. The presence of X decreased the polymerization rate 12-22% and the intrinsic η 4-fold. Halogen analyses showed that the proportion of allyl polymerization was 3.9 and 3.5 mole %, resp., in the absence and presence of X. The presence of X decreases the proportion of allyl type polymerization only slightly and the effect of the regulator on the mol. wt. appears to be the important factor which delays gelation. Effect of regulators on compn. of

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